FILE COVERS 1907 - 11 Jun 2008 VOL 148 ISS 24
FILE LAST UPDATED: 10 Jun 2008 (20080610/ED)

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L1   STRUCTURE UPLOADED

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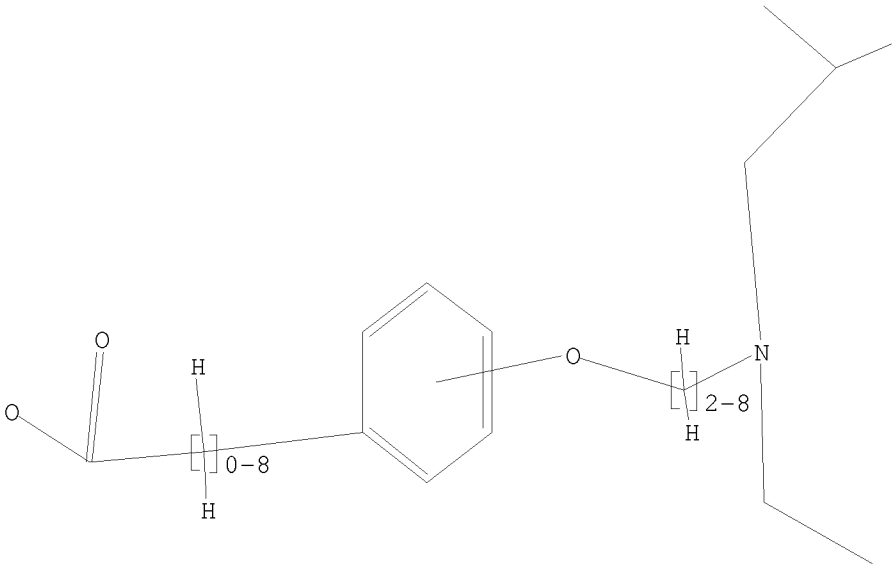
L1   STR



Structure attributes must be viewed using STN Express query preparation.

=> s l1 full
   REG1stRY INITIATED
Substance data SEARCH and crossover from CAS REGISTRY in progress...
Use DISPLAY HITSTR (or FHITSTR) to directly view retrieved structures.

FULL SEARCH INITIATED 16:10:40 FILE 'REGISTRY'
FULL SCREEN SEARCH COMPLETED -   43029 TO ITERATE

100.0% PROCESSED 43029 ITERATIONS 6 ANSWERS
SEARCH TIME: 00.00.01

L2 6 SEA SSS FUL L1

L3 0 L2

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L4 STRUCTURE UPLOADED

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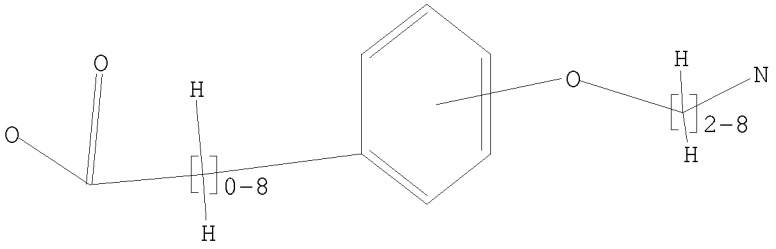
L4 STR



Structure attributes must be viewed using STN Express query preparation.

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Substance data SEARCH and crossover from CAS REGISTRY in progress...
Use DISPLAY HITSTR (or FHITSTR) to directly view retrieved structures.

FULL SEARCH INITIATED 16:11:50 FILE 'REGISTRY'
FULL SCREEN SEARCH COMPLETED - 2096300 TO ITERATE

 47.7% PROCESSED 1000000 ITERATIONS 1969 ANSWERS
INCOMPLETE SEARCH (SYSTEM LIMIT EXCEEDED)
SEARCH TIME: 00.00.13

FULL FILE PROJECTIONS: ONLINE **INCOMPLETE**
                        BATCH   **INCOMPLETE**
PROJECTED ITERATIONS: 2096300 TO 2096300
PROJECTED ANSWERS: 3935 TO 4319

L5 1969 SEA SSS FUL L4

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L6         296 L5

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=> s l6 full
L7         296 L5

=> s l7 and py<2002
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L8           73 L7 AND PY<2002

=> s l8 and phneyl?
              7 PHNEYL?
L9            0 L8 AND PHNEYL?

=> s l8 and phenyl?
         869610 PHENYL?
L10          25 L8 AND PHENYL?

=> d 1-25 ibib abs hitstr

L10  ANSWER 1 OF 25  CAPLUS  COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER:         1966:94262  CAPLUS
DOCUMENT NUMBER:          64:94262
ORIGINAL REFERENCE NO.:   64:17804g-h,17805a
TITLE:                    Catalyst mixtures for polyurethan reactions
INVENTOR(S):              Wild, James H.; Williams, Derek
PATENT ASSIGNEE(S):       Imperial Chemical Industries Ltd.
SOURCE:                   6 pp.
DOCUMENT TYPE:            Patent
LANGUAGE:                 Unavailable
FAMILY ACC. NUM. COUNT:   1
PATENT INFORMATION:

     PATENT NO.           KIND   DATE        APPLICATION NO.         DATE
     ---------------      ----   --------    --------------------    --------
     GB 1001458                  19650818    GB 1962-45463           19621203 <--
PRIORITY APPLN. INFO.:                       GB                      19621203
AB   The process for treating an organic compound containing two or more reactive
NCY
     radicals in which Y is O or S with a compound containing an active H is
     accelerated by the use of a mixture of quaternary ammonium, quaternary
     phosphonium, or ternary sulfonium salt of a strong acid, e.g. Bu3P(Me)I,
     PhCH2NMe3I, and Me3SI, and an organic metal composition of the type used as
     catalysts in polyurethan manufacturing  These quaternary or ternary salt
     catalysts are used from 0.05 to 5% by weight of the compound containing active
H.
     The catalysts are salts of acids whose pK value is <4 at 25°.  The
     preferred organic metal polyurethan catalyst compds. are Sn, Zn, or Pb
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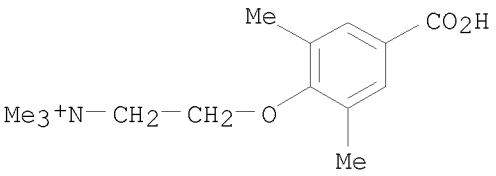
octanoate or Bu2Sn dilaurate.
IT 618880-92-5P, Ammonium, [2-[(4-carboxy-2,6-xylyl)oxy]ethyl]trimethyl, chloride
RL: PREP (Preparation)
(catalysts, in urethan polymer manufacture)
RN 618880-92-5 CAPLUS
CN Ethanaminium, 2-(4-carboxy-2,6-dimethylphenoxy)-N,N,N-trimethyl-, chloride (1:1) (CA INDEX NAME)

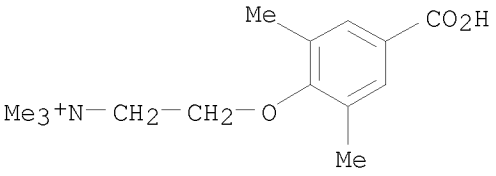


L10 ANSWER 2 OF 25 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 1966:20228 CAPLUS
DOCUMENT NUMBER: 64:20228
ORIGINAL REFERENCE NO.: 64:3778h,3779a-b
TITLE: Deactivation of catalyst residues in polyolefins
INVENTOR(S): Zikmund, Miroslav; Richtrova, Eva; Ambroz, Ludvik
SOURCE: 4 pp.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
CS 113620		19650215	CS	19630330 <--

PRIORITY APPLN. INFO.: CS 19630330
AB To a polyolefin heated to 20-100°, a solution of a mixture of alkyl- and arylammonium fluoroantimonates or plumbate (IV) salts of organic acids with an alkyl or arylammonium fluoride and (or) phenyl hydrazinium fluoride, in which alkyl is Me, Et, or Bu, and aryl is Ph or benzyl, in an organic solvent was added (concentration of fluorides 0.1-10 g./kg. of polymer and the weight ratio of plumbates to ammonium or hydrazinium salts was 1:1.2-1.5. Thus, polyethylene was prepared by polymerization in C7H16 with TiCl4 + Et2AlCl at 75°. The suspension of polyethylene was filtered to remove waxlike products and soluble catalyst components. The filtration cake was put in a C7H16 solution containing a 100% molar excess of a complex compound (SbF6)-(NEt4)+ based on Al and Ti in ash. The paste obtained was kept for 3 hrs. at 30° and then the polyethylene was filtered and washed with pure C7H16 and dried. The sheet (0.1 mm.) pressed from the product had good stability.
IT 618880-92-5, Ammonium, [2-[(4-carboxy-2,6-

xylyl)oxy]ethyl]trimethyl, chloride
(in catalyst removal from olefin polymers)
RN 618880-92-5 CAPLUS
CN Ethanaminium, 2-(4-carboxy-2,6-dimethylphenoxy)-N,N,N-trimethyl-, chloride (1:1) (CA INDEX NAME)



L10 ANSWER 3 OF 25 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 1964:477242 CAPLUS
DOCUMENT NUMBER: 61:77242
ORIGINAL REFERENCE NO.: 61:13494c-e
TITLE: Polymers and copolymers of azo dyes containing vinylsulfone groups
INVENTOR(S): Grafmueller, Fritz; Weissermel, Klaus
PATENT ASSIGNEE(S): Farbwerke Hoechst A.-G.
SOURCE: 5 pp.; Addn. to Ger. 1,129,697 (CA 57, 7473b)
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 1173652		19640709	DE 1961-F35394	19611121 <--

PRIORITY APPLN. INFO.: DE 19611121

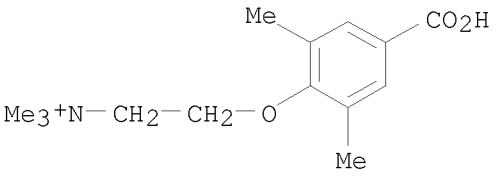
AB High-mol.-weight polymers were prepared by solution or suspension polymerization of vinylsulfone group-containing azo dyes of the formula H2C:CHSO2AN:NRNH2, in which A is an aryl radical which may be substituted in the nucleus by an alkyl or hydroxy alkyl group or a halogen atom, and R is mono- or polysubstituted aryl, pyrazolone, or acetoacetylarylamide radical, in the presence of 0.1-5.0 weight% of an anionic catalyst, based on the weight of the monomer(s), or by the eopolymerization of such dyes with other anionic-polymerizable monomers. E.g., benzyltrimethylammonium hydroxide 0.06 in pyridine 2 was added dropwise to 4-aminophenylvinyl sulfone (I) 20 and 4-vinylsulfonyl-2'-methyl-4'-aminoazobenzene (II) 0.5 in pyridine 40 parts. Polymerization set in shortly. During polymerization, the temperature rose from 20 to 50° in spite of cooling, and the mixture became highly viscous. After 3 hrs., the mixture was stirred into MeOH. The copolymer accumulated as a finely divided, bright-red powder. The monomers were removed by extracting the mixture for 24 hrs. (both monomers were MeOH-soluble), and the mixture dried at 70° to yield 20 parts by weight copolymer. The copolymer began to sinter at 170° and changed into

a thermoplastic mass at 195-210°, from which filaments could be drawn. It was soluble in HCONMe2, Me2SO, and α-butyrolactone; the reduced viscosity was 0.08 (in HCONMe2 at 25°). The color of the polymeric dyes corresponds to that of the monomeric dyes. The reactive basic homo- and copolymers can be used for coloring resins, especially polyesters, polyamides, and polyacetals. They are very heat- and moisture-resistant.

IT 618880-92-5, Ammonium, [2-[(4-carboxy-2,6-xylyl)oxy]ethyl]trimethyl, chloride
(catalysts, in polymerization of azo dyes with vinylsulfone groups)

RN 618880-92-5 CAPLUS

CN Ethanaminium, 2-(4-carboxy-2,6-dimethylphenoxy)-N,N,N-trimethyl-, chloride (1:1) (CA INDEX NAME)



L10 ANSWER 4 OF 25 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1964:91269 CAPLUS

DOCUMENT NUMBER: 60:91269

ORIGINAL REFERENCE NO.: 60:15986f-h

TITLE: Asymmetric synthesis of polymers obtained by cationic processes

AUTHOR(S): Natta, Giulio; Farina, Mario; Peraldo, Mario; Bressan, Giancarlo

CORPORATE SOURCE: Politecnico, Milan

SOURCE: Chem. Ind. (Milan) (1961), 43(2), 161-2

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

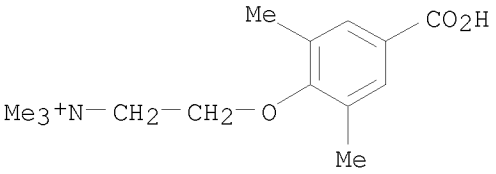
AB Benzofuran was polymerized to optically active polybenzofuran (I) by a cationic mechanism. Temps. of -80 to -100° and toluene solvent were used with asym. catalysts, e.g. alkylaluminum halides with optically active acids, alcs., hydroxy acids, amino acids, quaternary ammonium salts, alkaloids, or terpenes. I prepared as above by EtAlCl2 (II) and (-)-β- phenylalanine, had an intrinsic viscosity (toluene, 30°) = 0.6 dl./g., [α]D (2.0% C6H6) = -33.1, [M]D = -39.1 (referred to the monomeric unit), and [M]303 (dioxane) = - 800. I prepared by II and (-)-brucine had [α]D = +2.8. I prepared by II and (+)-camphorsulfonic acid had [α]D = -3.6. Infrared examination gave a structure for I in which all the C atoms of the chain are asym. I was amorphous on x-ray examination, but is believed to have a head-to-tail and diisotactic structure. The difficulty of crystallization of I is tentatively attributed to steric hindrance. The absence of optically active end groups derived from the catalyst was shown by infrared measurements and the use of 35S-labeled cocatalysts. Optical activity is considered to be

induced in I by an asym. counterion.

IT 618880-92-5, Ammonium, [2-[(4-carboxy-2,6-xylyl)oxy]ethyl]trimethyl, chloride
(catalysts from Al compds. and optically-active, in asymmetric polymerization of benzofuran)

RN 618880-92-5 CAPLUS

CN Ethanaminium, 2-(4-carboxy-2,6-dimethylphenoxy)-N,N,N-trimethyl-, chloride (1:1) (CA INDEX NAME)



L10 ANSWER 5 OF 25 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1964:61387 CAPLUS

DOCUMENT NUMBER: 60:61387

ORIGINAL REFERENCE NO.: 60:10819a-c

TITLE: Catalysts for polymerization of ethylene and propylene

PATENT ASSIGNEE(S): Solvay & Cie

SOURCE: 7 pp.

DOCUMENT TYPE: Patent

LANGUAGE: Unavailable

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
BE 624645		19630509	BE	<--
GB 960086			GB	
PRIORITY APPLN. INFO.:			NL	19611115

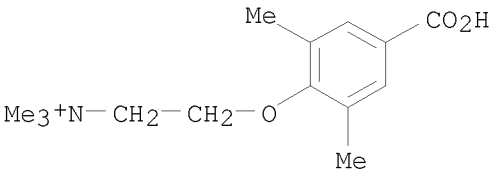
AB Relatively low mol. wts. of 30,000-40,000 are achieved by addition of amines or quaternary ammonium salts to the ternary catalyst (Ziegler type) which consists of: (a) a metal, metal hydride, or an organometallic composition of metals of Groups IV, V, or VI; (b) a compound of a multivalent metal with at least 3 valences; and (c) a halide of an element of Group III or V. For example, (a) may be Bu4Sn, (b) TiCl4, and (c) AlCl3. The amines include Pr2NH, PhNH2, pyridine, N,N'-diphenyl-p-phenylenediamine, naphthylamine, hexylamine, diphenylguanidine, and sym- or N,N-diethyl-p-phenylenediamine. The quaternary ammonium salts used should be dimethylbenzyllaurylammonium, trimethylbenzylammonium, dodecyltrimethylammonium, or octadecyltrimethylammonium chloride, or tetrabutylammonium iodide. Amts. of the addns. vary between 0.01 and 1 mole per g.-atom of the multivalent metal with 3 valencies. For example, C2H4 is polymerized for comparison either with the TiCl4Bu4Sn-AlCl3 ternary catalyst or with addns, of 1 of the above amines. Thus, a catalyst is prepared by warming at 25° for 48 min. TiCl4 184, Bu4Sn 708, and AlCl3 245 mg. A suspension of the catalyst is diluted with 1 l. of dry, pure

C6H14. The solution is poured into an autoclave heated to 80° and C2H4 is introduced at 10 atmospheric at a flow rate of 120 g./hr. The polymerization is stopped after 2 hrs. The polyethylenes are washed, dried, and examined The mol. weight is ascertained by a viscosimetric method. Polymerization without the amine addition gives a polyethylene of mol. weight 55,000; with addition of 20.0 mg. hexylamine/l. C6H14, the mol. weight is only 37,000.

IT 618880-92-5, Ammonium, [2-[(4-carboxy-2,6-xylyl)oxy]ethyl]trimethyl, chloride
(catalysts, in polymerization of C2H4 and propene, for mol. weight control)

RN 618880-92-5 CAPLUS

CN Ethanaminium, 2-(4-carboxy-2,6-dimethylphenoxy)-N,N,N-trimethyl-, chloride (1:1) (CA INDEX NAME)



L10 ANSWER 6 OF 25 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1963:408695 CAPLUS

DOCUMENT NUMBER: 59:8695

ORIGINAL REFERENCE NO.: 59:1531d-h,1532a-d

TITLE: Quaternary ammonium salts from tertiary 2-phenoxyethylamines

INVENTOR(S): Copp, Frederick C.; Elphick, Albert R.; Coker, Geoffrey G.

PATENT ASSIGNEE(S): Wellcome Foundation Ltd.

SOURCE: 13 pp.

DOCUMENT TYPE: Patent

LANGUAGE: Unavailable

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
GB 919126		19630220	GB	19580701 <--

PRIORITY APPLN. INFO.: GB 19580701

GI For diagram(s), see printed CA Issue.

AB (Phenoxyalkyl)dialkylamines are treated with alkyl halides to give I and II, where R and R1 are Me or Et, R2 and R3 are H, halogen, MeO, or Me, Y is NO2, Cl, an alkyl, or an alkoxy group, Z is a C1-3 alkoxy group, and X is iodine or Br; I and II can be used as depressants for the peripheral sympathetic nervous system. Thus, 136 g. 4-hydroxy-3,5-dimethylbenzophenone is added to a solution of 13.8 g. Na in 950 mL. hot EtOH, 136 g. BrCH2CH2Br added, the mixture refluxed 7 h., .apprx.700 mL. EtOH evaporated in vacuo, the residue poured into 500 mL. H2O, the oil that

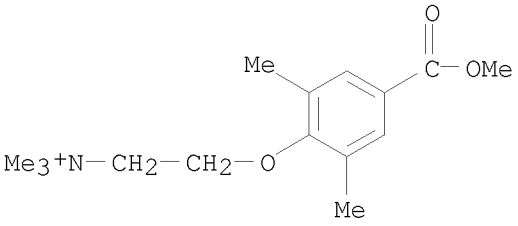
sep. extracted with Et2O, the extract washed with 5N NaOH, the Et2O evaporated, and
the residue distilled to give 2-(4-benzoyl-2,6-dimethylphenoxy)ethyl bromide (III), b0.01 182-6°, m.p. 76°. A mixture of 16.7 g. III and 50 g. 25% Me2NH(MeOH) is heated in a sealed tube at 100° 6 h., the mixture evaporated, excess 5N NaOH added to the residue, the oil that sep. extracted with Et2O, the Et2O evaporated, and the residue distilled to give 1-(4-benzoyl-2,6-dimethylphenoxy)-2-dimethylaminoethane (IV), b0.001 162-7°. MeI (4 g.) is added to a solution of 4 g. IV in Me2CO, the mixture kept 1 h., refluxed 30 min., and cooled to give N-[2-(4-benzoyl-2,6-dimethylphenoxy)ethyl]-N,N,N-trimethylammonium iodide, m. 208-9° (EtOH). Similarly prepared are I (Y, R2, R3, R, R1, X, m.p. given): H, Me, Me, Me, Et, iodine, 185-6° (EtOH); H, Me, Me, Me, Me, Br, 204-5° (iso-PrOH); p-Me, Me, Me, Me, Me, Br (hemihydrate), 216-17° (EtOH-iso-PrOH); m-Me, Me, Me, Me, Me, Br, 221°; o-Cl, Me, Me, Me, Me, Br, 204-5°; m-Cl, Me, Me, Me, Me, Br, 203-4°; p-Cl, Me, Me, Me, Me, Br, 226-7°; o-MeO, Me, Me, Me, Me, Br, 216-17°; m-MeO, Me, Me, Me, Me, Br, 176-8°; p-MeO, Me, Me, Me, Me, Br, 189-90°; p-EtO, Me, Me, Me, Me, Br, 203°; p-NO2: Me, Me, Me, Me, Br, 240-1°; H, Cl, Cl, Me, Me, Br, 186°, H, H, H, Me, Me, Br, 196-7°; p-NH2, Me, Me, Me, Me, iodine, 239-41°; H, H, Br, Me, Me, iodine, 209-10° (MeOH); H, H, Br, Me, Et, iodine, 165-6°; H, H, Cl, Me, Me, Br, 199-200° (iso-PrOH-Et2O); H, H, F, Me, Me, iodine, 227-80°; H, H, F, Me, Et, iodine (hemihydrate), 211-12°; H, Br, Me, Me, Me, iodine, 178-9° (EtOH-iso-PrOH); H, Me, Et, Me, Et, iodine, 221-2°; H, Me, Me, Me, HO(CH2)2, iodine, 160-1° (EtOH); H, Me, Me, HO(CH2)2, HO(CH2)2, iodine, 110-11°; H, Me, Me, Et, Et, iodine, 149-50° (EtOH); H, H, MeO, Me, Me, iodine, 189-90° (EtOH-ether); H, Me, Me, Me, Me, Cl (hydrate), 209° (iso-PrOH-Et2O); and H, Me, Me, Me, Me, MeSO4, 138-9° (EtOH-EtOAc). Similarly prepared are II (Z, R2, R3, R, R1, X, m.p. given): Me, Me, Me, Me, Me, iodine, 182-3° (EtOH); Et, Me, Me, Me, Me, iodine, 181-2° (EtOH); Et, Me, Me, Me, Et, Br, 109-11° (iso-PrOH-Et2O); PhCH2, Me, Me, Me, Me, Br, 148-50° (iso-PrOH); EtO, H, H, Me, Me, iodine, 157-60° (EtOAc-EtOH); MeO, H, H, Me, Me, iodine, 205-7° (Me2CO-EtOAc); MeO, Me, H, Me, Me, iodine, 149-51° (EtOH-EtOAc); MeO, Me, Me, Me, Me, iodine, 213-15° (EtOH-EtOAc); EtO, H, H, Et, Et, iodine, 128° (EtOH-EtOAc); EtO, Me, H, Me, Me, iodine, 163-5° (EtOH-EtOAc); iso-PrO, Me, Me, Me, Me, iodine, 186-7° (iso-PrOH); MeO, MeO, H, Me, Me, iodine 181-4° (EtOH); EtO, MeO, H, Me, Me, iodine, 136-8° (EtOH); EtO, MeO, MeO, Me, Me, iodine, 208-10° (EtOH); MeO, Br, H, Me, Me, iodine, 196-9° (EtOH); MeO, Br, H, Me, Et, iodine, 186-9° (EtOH); EtO, Br, H, Me, Me, iodine, 184-5° (iso-PrOH); EtO, Br, H, Me, Et, iodine, 121-4° (iso-PrOH); and EtO, Me, Me, Me, Me, iodine, 177-9° (EtOH-EtOAc). Also prepared are (m.p. given) N-[3-(4-benzoyl-2,6-dimethylphenoxy)propyl]-N,N,N-trimethylammonium bromide, 160-1°; N-[2-(4-benzoyl-2,6-dimethylphenoxy)-1-methylethyl]-N,N,N-trimethylammonium iodide, 215-16° (EtOH); N-[2-(4-benzoyl-2,6-dimethylphenoxy)-2-methylethyl]-N,N,N-trimethylammonium iodide, 167° (EtOH); N-[2-(4-benzoyl-3-hydroxyphenoxy)ethyl]-N,N,N-trimethylammonium iodide, 139-40° (EtOH); N-[2-(4-acetamido-2,6-dimethylphenoxy)ethyl]-N,N,N-trimethylammonium iodide, 242-4° (MeOH); and N-[2-(4-propionylamino-

2,6-dimethylphenoxy)ethyl]-N,N,N-trimethylammonium iodide, 197-9° (EtOH).

IT 701193-78-4P, Ammonium, [2-[(4-carboxy-2,6-xylyl)oxy]ethyl]trimethyl, Me ester 805949-72-8P, Ammonium, [2-[(4-carboxy-2,6-xylyl)oxy]ethyl]trimethyl, iso-Pr ester 875831-55-3P, Benzoic acid, 4-[2-(dimethylamino)ethoxy]-3,5-dimethoxy-, isopropyl ester
RL: PREP (Preparation)
(preparation of)

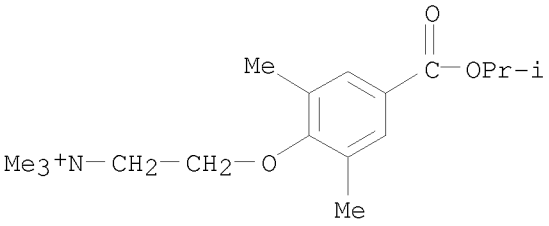
RN 701193-78-4 CAPLUS

CN Ethanaminium, 2-[4-(methoxycarbonyl)-2,6-dimethylphenoxy]-N,N,N-trimethyl- (CA INDEX NAME)



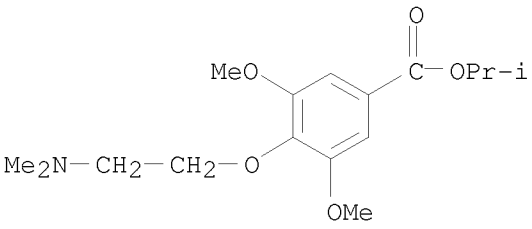
RN 805949-72-8 CAPLUS

CN Ethanaminium, 2-[2,6-dimethyl-4-[(1-methylethoxy)carbonyl]phenoxy]-N,N,N-trimethyl- (CA INDEX NAME)



RN 875831-55-3 CAPLUS

CN Benzoic acid, 4-[2-(dimethylamino)ethoxy]-3,5-dimethoxy-, 1-methylethyl ester (CA INDEX NAME)

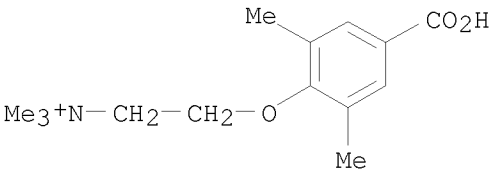


L10 ANSWER 7 OF 25 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1963:82225 CAPLUS
DOCUMENT NUMBER: 58:82225
ORIGINAL REFERENCE NO.: 58:14148f
TITLE: Cyanoethyl polyamides
PATENT ASSIGNEE(S): Romania, Ministry of Petroleum and Chemical Industry
SOURCE: 2 pp.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
GB 920213		19630306	GB 1959-23855	19590710 <--
PRIORITY APPLN. INFO.:			RO	19580712

AB Polyamides are modified by treatment at 20-90° with acrylonitrile (I) in the presence of basic catalysts. Thus, a suspension of 0.4 g. powdered NaOH and 4 g. powdered polycaprolactam (II) in a solution of 10 g. I (stabilized with 0.5% phenyl-β-naphthylamine) in 50 cc. dioxane was heated at 75-7° for 1 hr. Working up resulted in 8.5 g. yellowish powder containing 6.5% nitrile N, 16.3% total N, and 70% cyanoethyl-substituted polyamide units.
IT 618880-92-5, Ammonium, [2-[(4-carboxy-2,6-xylyl)oxy]ethyl]trimethyl, chloride
(catalysts, in cyanoethylation of polyamides)
RN 618880-92-5 CAPLUS
CN Ethanaminium, 2-(4-carboxy-2,6-dimethylphenoxy)-N,N,N-trimethyl-, chloride (1:1) (CA INDEX NAME)



● Cl⁻

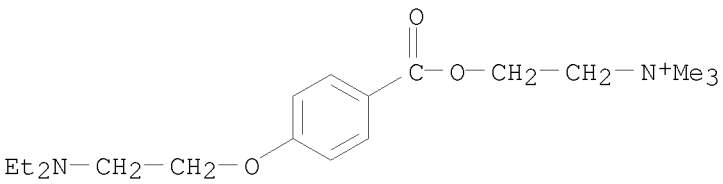
L10 ANSWER 8 OF 25 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 1963:21714 CAPLUS
DOCUMENT NUMBER: 58:21714
ORIGINAL REFERENCE NO.: 58:3633e-f
TITLE: Relations between structure and albumin-binding of amines tested with crossing-paper electrophoresis
AUTHOR(S): Bickel, M. H.; Bovet, D.
CORPORATE SOURCE: Ist. Super. Sanita, Rome
SOURCE: Journal of Chromatography (1962), 8, 466-74
CODEN: JOCRAM; ISSN: 0021-9673
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
AB cf. CA 56, 4041h. A total of 75 N-containing substances was screened with regard to their interaction with blood albumin by means of crossing-paper

electrophoresis (loc. cit.). Only tertiary amines with at least 1 substantial radical interact, whereas primary and secondary amines and quaternary NH4+ salts do not. With mixed amines, interaction only occurs if the tertiary N dominates the other amino groups.

IT 856619-26-6, Choline, p-[2-(diethylamino)ethoxy]benzoate (ester) (reaction with albumin)

RN 856619-26-6 CAPLUS

CN Ethanaminium, 2-[[4-[2-(diethylamino)ethoxy]benzoyl]oxy]-N,N,N-trimethyl- (CA INDEX NAME)



L10 ANSWER 9 OF 25 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1963:21330 CAPLUS

DOCUMENT NUMBER: 58:21330

ORIGINAL REFERENCE NO.: 58:3570c

TITLE: Vinyl polymer compositions for dentures

INVENTOR(S): Rossetti, Carlo

PATENT ASSIGNEE(S): Kulzer & Co. G.m.b.HM.

SOURCE: 3 pp.

DOCUMENT TYPE: Patent

LANGUAGE: Unavailable

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 1138226		19621018	DE 1953-C7820	19530629 <--

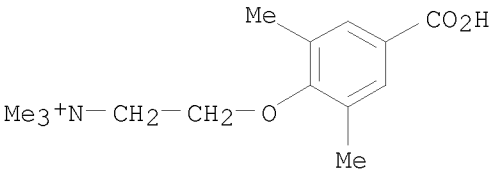
PRIORITY APPLN. INFO.: DE 19530629

AB Polymers suitable for artificial teeth, fillings, etc., are prepared by mixing a vinyl monomer, a powdered polymer, a min. amount of a sulfinic acid, and a quaternary base. Thus, to monomeric Me methacrylate containing 2% benzenesulfinic acid, 0.5% benzyl(diisobutylphenoxyethoxy)-dimethylammonium hydroxide was added. Enough powdered poly(Me methacrylate) was added to make a readily workable paste. Polymerization was complete at 18° after 8 min.

IT 618880-92-5, Ammonium, [2-[(4-carboxy-2,6-xylyl)oxy]ethyl]trimethyl, chloride (catalysts from sulfinic acids and, in polymerization of Me methacrylate for dentures)

RN 618880-92-5 CAPLUS

CN Ethanaminium, 2-(4-carboxy-2,6-dimethylphenoxy)-N,N,N-trimethyl-, chloride (1:1) (CA INDEX NAME)



● Cl⁻

L10 ANSWER 10 OF 25 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 1962:456059 CAPLUS
DOCUMENT NUMBER: 57:56059
ORIGINAL REFERENCE NO.: 57:11115c-f
TITLE: Basic substituted alkyl ethers from o-cresotic acid esters and its salts
INVENTOR(S): Hiltmann, Rudolf; Mietzech, F.; Mietzsch, Fritz; Kaemmeter, Kurt
SOURCE: 4 pp.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

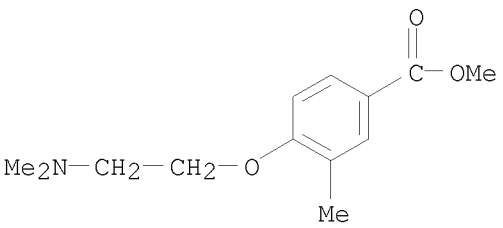
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 1118219		19621130	DE 1957-F0022404	19570221 <--

PRIORITY APPLN. INFO.: DE 19570221

AB Anesthetics for veterinary use with prolonged efficiency are prepared by reaction of o-cresotic acid esters with dialkylaminoalcs, or HORY (R = alkylene with 2 or 3 C atoms, Y = a substituent transformable into mono- or dialkylamino group) in presence of acid binding agents. E.g., 41.5 g. 3,2-Me(HO)C6H3CO2Me is added to 5.8 g. Na in 200 ml. MeOH, distd, in vacuo. Dry residue is suspended in 200 ml. anhyd, toluene, boiled and 30 g. Me2NCH2Cl, diluted with PhMe is dropped in slowly and refluxed 24 hrs. After cooling, the solution is washed with H2O, 2 times with 5% NaOH. After extraction with 2N HCl, base is precipitated with K2CO3 solution, taken up in C6H6, dried, and distilled, giving 30 g. 3,2-Me(Me2NCH2CH2O)C6H3CO2Me, b5 1346°; HCl salt m. 127°. Similarly were prepared: 3,2-Me(Me2NCH2CH2CH2O)C6H3CO2Me, b5 149-52° (HCl salt m. 90-1°); 3,2-Me(Et2NCH2CH2O)C6H3CO2Me, b4 147-9° (HCl salt m. 122°); 3,2-Me(Me2NCH2CH2CH2O)C6H3CO2Et, b4 145-9° (HCl salt m. 143-4°); 3,2-Me(Et2NCH2CH2CH2O)C6H3CO2Et, b3 161-2°; 3,2-Me2NCH2CH2O)C6H3CO2Et, b5 151° phosphate m. 93-5°.

IT 857370-73-1P, m-Toluic acid, 4-[2-(dimethylamino)ethoxy]-, methyl ester, hydrochloride
RL: PREP (Preparation)
(preparation of)

RN 857370-73-1 CAPLUS
CN Benzoic acid, 4-[2-(dimethylamino)ethoxy]-3-methyl-, methyl ester, hydrochloride (1:1) (CA INDEX NAME)



L10 ANSWER 11 OF 25 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 1962:430619 CAPLUS
DOCUMENT NUMBER: 57:30619
ORIGINAL REFERENCE NO.: 57:6178c-g
TITLE: Antistatic, soft, and microorganism-resistant fabric
INVENTOR(S): Sherrill, Joseph C.; Linfield, Warner M.; Marsh, Byron E.
PATENT ASSIGNEE(S): Armour & Co.
SOURCE: 5 pp.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 3033704		19620508	US 1959-814149	19590519 <--
DE 1195265			DE	
GB 930333			GB	

GI For diagram(s), see printed CA Issue.

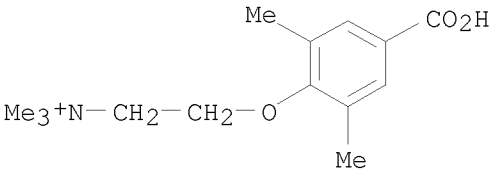
AB A laundered fabric is impregnated, while rinsing, with one or more cationic surfactants (I) and an organomercurial germicide (II), to render it antistatic, soft and partially free from microorganisms. The fabric is then dried. Three formulas for I are specified: [R1N(R2)(R3)2]+X- (III), [(R1)2N(R2)R3]+X- (IV), and V where R1 is a C10-22 alkyl radical, R2 is a benzyl radical or an alkyl radical containing <3 C atoms, R3 is an alkyl radical containing <3 C atoms, and X is chloride, bromide, sulfate, or an alkyl sulfate in which the alkyl radical contains <5 C atoms. R1 may be a natural mixture derived from tallow, soybean, or coconut oil. III tends toward greater germicidal activity than IV, but the latter has greater softening action and even better results are obtained from III and IV, in which R1 is a C12-18 alkyl radical, R2 is a benzyl radical, R3 a Me radical, and X is chloride. Best results are obtained when II is phenylmercuric acetate, propionate, butyrate, chloride, bromide, or iodide. A typical formulation is 13.7% Softener 2-132 (75%), 10% Arquad S (50%), 0.85% PhHg-OCOC2H5, 2% hexylene glycol, 0.2% Na2SO4, 0.5% pigment dye, 0.38% brightener, 0.125% perfume, and H2O up to 100%. This is added to the rinse at 12 fl. oz./100 lb. fabric. An example of the efficacy of the treatment is shown, wherein a fabric treated with a concentration of 0.079% of I and 50 p.p.m. II, based on the weight of fabric, shows an average

zone of inhibition vs. Staphylococcus aureus of 6 mm. Where treatment takes place in 2 stages, i.e. in a solution of I and then in a solution of II, the zone of inhibition is narrower.

IT 618880-92-5, Ammonium, [2-[(4-carboxy-2,6-xylyl)oxy]ethyl]trimethyl, chloride
(as cationic surfactant in antistatic, bacteriostatic softening finish for textiles)

RN 618880-92-5 CAPLUS

CN Ethanaminium, 2-(4-carboxy-2,6-dimethylphenoxy)-N,N,N-trimethyl-, chloride (1:1) (CA INDEX NAME)



L10 ANSWER 12 OF 25 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1962:430499 CAPLUS

DOCUMENT NUMBER: 57:30499

ORIGINAL REFERENCE NO.: 57:6154d-g

TITLE: Organopolysiloxane foam preparation at room temperature

INVENTOR(S): Weyer, Donald E.

PATENT ASSIGNEE(S): Dow Corning Corp.

SOURCE: 4 pp.

DOCUMENT TYPE: Patent

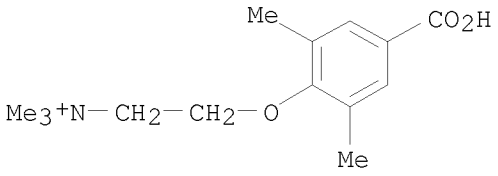
LANGUAGE: Unavailable

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 3024210		19620306	US 1959-853697	19591118 <--

AB A permanent, heat-stable foam, created by rapid evolution of H, is formed at room temperature by mixing an organopolysiloxane, catalyst, and a hydroxylated compound The organopolysiloxane of the general formula (RHSiO)x contains 1-1.8 hydrocarbon radicals per Si which can be either univalent hydrocarbon, halogenated hydrocarbon, or halophenoxymethyl radicals. In addition, the organopolysiloxane contains at least 1% by weight of units with at least 1 H atom attached to Si. Often copolymers or mixts. of homopolymers are used. The catalysts are quaternary ammonium compds. of the type R4'NOH, R4'NOR'', R4'NOCOR''', and R3SiONR4' where R', R'', and R''' are mainly aliphatic radicals. The hydroxylated compound can be a low-mol.-weight silanol, H2O, or alc. In an example, 100 g. of a copolymer of phenylmethylsiloxane 40, methylhydrogensiloxane 20, monophenylsiloxane 30 and HSiO3/2 10 mole % were mixed with 2 g. BuOH and 2 cc. of a 20% solution of benzyl(β-hydroxyethyl)dimethylammonium

butoxide. Foaming was complete within 0.5 hr.; foam d. 25 lb./cu. ft.
IT 618880-92-5, Ammonium, [2-[(4-carboxy-2,6-xylyl)oxy]ethyl]trimethyl, chloride
(catalysts, in foaming of polysiloxanes in presence of hydroxy compds.)
RN 618880-92-5 CAPLUS
CN Ethanaminium, 2-(4-carboxy-2,6-dimethylphenoxy)-N,N,N-trimethyl-, chloride (1:1) (CA INDEX NAME)



● Cl−

L10 ANSWER 13 OF 25 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 1962:2737 CAPLUS
DOCUMENT NUMBER: 56:2737
ORIGINAL REFERENCE NO.: 56:565d-f
TITLE: Selective coating of surfaces with organopolysiloxane resins
INVENTOR(S): Clark, Harold A.
PATENT ASSIGNEE(S): Dow Corning Corp.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 3002848		19611003	US 1960-669060	19600204 <--

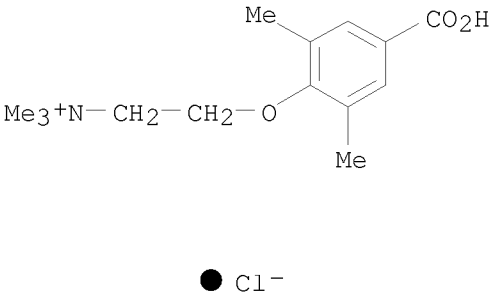
PRIORITY APPLN. INFO.: US 19600204
AB A method is described for selectively coating surfaces with organopolysiloxane resins which gives a sharp delineation between the coated and uncoated portions of the surface and provides an improved way of preparing electronic equipment. Thus, a com. Cu-coated epoxide resin-glass laminate was dipped into a 50% toluene solution of a copolymer of 75 mole % monoethylsiloxane and 25 mole % mono(2-phenylpropyl)siloxane, containing 1.25% by weight Si-bonded OH and 0.15% by weight benzyltrimethylammonium acetate, based on the weight of the copolymer. The coated laminate was dried at room temperature to remove the solvent. A trimethylolethane isophthalate ester (acid number 16) was dissolved in a mixture of BuOAc and EtOH to give a 50% by weight solution of the ester which was applied to various areas of the uncured silicone resin coating on the Cu surface. The BuOAc-EtOH solvent was evapd, at room temperature, and the assembly cured 20 min. at 150°. The laminate was washed with Me Cellosolve which removed the ester coating, with the uncured silicone resin beneath the coating leaving a sharply defined pattern corresponding

to the areas covered by the acid ester. The exposed Cu surface was etched with a standard FeCl3-HCl solution which did not affect the Cu under the cured siloxane resin. The cured resin was removed by washing with toluene which exposed a clean Cu surface ready for fabrication of electronic devices.

IT 618880-92-5P, Ammonium, [2-[(4-carboxy-2,6-xylyl)oxy]ethyl]trimethyl, chloride
RL: PREP (Preparation)
(catalysts, in curing of siloxanes in manufacture of printed elec. circuits)

RN 618880-92-5 CAPLUS

CN Ethanaminium, 2-(4-carboxy-2,6-dimethylphenoxy)-N,N,N-trimethyl-, chloride (1:1) (CA INDEX NAME)



L10 ANSWER 14 OF 25 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 1958:40409 CAPLUS
DOCUMENT NUMBER: 52:40409
ORIGINAL REFERENCE NO.: 52:7216b-i,7217a
TITLE: Synthetic curare compounds. VIII. Ether-esters of choline with p-hydroxyaryl- and arylalkylcarboxylic acids
AUTHOR(S): Rosnati, Vittorio
SOURCE: Rend. ist. super. sanita (1955), 18, 998-1013
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

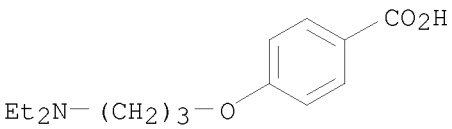
AB p-IR'3N(CH2)2O2CC6H4O(CH2)nNR3I (I), p-IR'3(CH2)2O2CCH2C6H4O(CH2)nNR3I (II), and p-IR'3N(CH2)2O2CCH2CH2C6H4O(CH2)nNR3I (III) were prepared, n = 2 or 3, and R, R' = Me or Et. Several of the intermediates prepared were new. I were prepared in fair to good yields through the following steps. Et p-hydroxybenzoate in absolute EtOH containing Na refluxed with Cl(CH2)nNR2, NaCl filtered off, the filtrate evaporated, extracted with Et2O, and distilled gave p-R2N(CH2)nOC6H4CO2Et (IV). So obtained were IV (R = Me, n = 3), b0.05 119-20°, and IV (R = Et, n = 3), b0.06 128-9°. From IV, the intermediate p-R2N(CH2)nOC6H4.CO2(CH2)2NR'2 (V) resulted by transesterification with excess HO(CH2)2NR2 and a small amount of Na, or in the case of IV (R = Me, n = 3) (which failed to react) by saponifying the Et ester to the free acid, treating the dried acid with PCl5 to form HCl.Me2N(CH2)3OC6H4COCl, which was in turn reacted with excess HO(CH2)2NMe2 in CHCl3 to form V (R, R' = Me, n = 3), b0.07 146-9°, V (R = Et, R' = Me, n = 3), b0.06 133-4°, V (R, R' = Et, n = 3), b0.05 152-4°. These were treated with MeI or EtI to form I: R3,

R'3 = Me3, n = 3 (VI), m. 262-4°; R3 = Et2Me, R'3 = Me3 (VII), m. 227-8°; R3, R'3 = Et3 (VIII), m. 197-9°. II were similarly prepared from p-hydroxyphenylacetic acid through p-R2N(CH2)nOC6H4CH2CO2Et (IX): R = Et, n = 2, b0.06 116-18°; R = Me, n = 3, b0.05 113-14°; R = Et, n = 3, b0.06 132°. From IX, further reaction with HO(CH2)nNR2 yielded p-R2N(CH2)nOC6H4CH2CO2(CH2)2NR'2 (X): R, R' = Et, n = 2, b0.06 142-5°; R, R' = Me, n = 3, b0.06 127-9°; R = Et, R' = Me, n = 3, b0.06 159-60°; R, R' = Et, n = 3, b0.06 162-3°. X with MeI or EtI yielded II in fair to good yields: R3, R'3 = Et3, n = 2 (XI), viscous oil; R3, R'3 = Me3, n = 3 (XII), m. 142-4°; R3 = Et2Me, R'3 = Me3 (XIII), viscous oil; R3, R'3 = Et3 (XIV), viscous oil. III (R3, R'3 = Et3) (XV), m. 159°, was prepared from 3-(p-hydroxyphenyl)propionic acid via p-Et2N(CH2)2OC6H4(CH2)2CO2Et, b0.5 150-2°, and p-Et2N(CH2)2OC6H4(CH2)2CO2(CH2)2NEt2, b0.07 174-6°. Among the curarizing agents tested, XII and XIII were not effective (at 0.05 mg./kg.), gave action of very brief duration, and were relatively low in toxicity. VI and VII were also quite effective (at 0.2 mg./kg.) with a more prolonged action similar to that of Flaxedil. The others (VIII, XI, XIV, XV) were less effective, with XIV and XV lowest in toxicity. Other compds. prepared were: p-MeO2CCH2OC6H4CO2Me, m. 96-8°, by refluxing 30 g. p-carboxyphenoxyacetic acid (XVI) (cf. Christiansen, C.A. 19, 1417) with 150 ml. MeOH saturated with HCl 7 hrs., filtering, and crystallizing the solution on ice (yield 22.5 g.). p-ClOCCH2OC6H4COCl (XVII), b0.08 107-8°, was prepared from 20 g. XVI by adding 40 g. PCl5 in small portions, allowing the reaction to subside, refluxing 1 hr., extracting the material with C6H6, and distilling p-Me2N(CH2)2O2CCH2OC6H4CO2(CH2)2NMe2 (XVIII), b0.05 160-78°, was prepared in 6.6 g. yield by dissolving 10 g. HO(CH2)2NMe in 150 ml. CHCl3, saturating the solution with HCl gas, adding 8 g. XVII in 60 ml. CHCl3, refluxing 6 hrs., cooling, adding 50 ml. ice H2O, acidifying with 1:1 HCl, removing the CHCl3 phase, neutralizing the aqueous phase with K2CO3, and extracting with Et2O. XVIII with MeI yielded IMe3N(CH2)2O2CCH2OC6H4CO2(CH2)2NMe3I, m. 231-3°. The Et analog of XVIII, b0.06 171-3°, was made in a similar way, but reaction with EtI yielded p-IEt3N(CH2)2O2CC6H4OCH2CO2H, m. 149-51°, which crystallized by slowly adding Et2O to the cold EtOH solution Dimethylaminoethyl phenoxyacetate, b0.6 109-10°, its Et analog, b0.4 115-16°, and the respective quaternary compds., m. 147°, and m. 140-1°, were prepared in similar fashion from ClOCCH2OPh and the HCl salt of the amino alc.

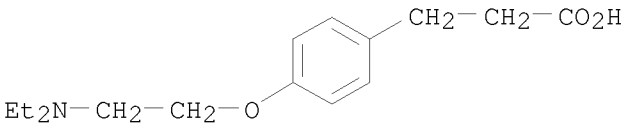
IT 551935-15-0, Benzoic acid, p-(3-diethylaminopropoxy)- 856639-07-1, Hydrocinnamic acid, p-(2-diethylaminoethoxy)- 857169-86-9, Acetic acid, [p-(3-dimethylaminopropoxy) phenyl]- 857170-47-9, Acetic acid, [p-(3-diethylaminopropoxy)phenyl]- (derivs.)

RN 551935-15-0 CAPLUS

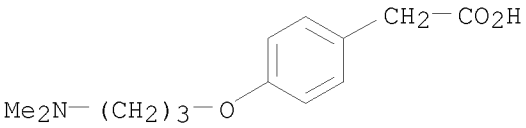
CN Benzoic acid, 4-[3-(diethylamino)propoxy]- (CA INDEX NAME)



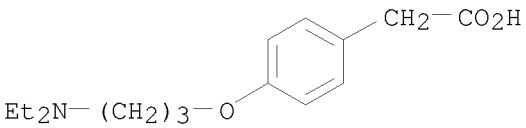
RN 856639-07-1 CAPLUS
CN Benzenepropanoic acid, 4-[2-(diethylamino)ethoxy]- (CA INDEX NAME)



RN 857169-86-9 CAPLUS
CN Benzeneacetic acid, 4-[3-(dimethylamino)propoxy]- (CA INDEX NAME)



RN 857170-47-9 CAPLUS
CN Benzeneacetic acid, 4-[3-(diethylamino)propoxy]- (CA INDEX NAME)



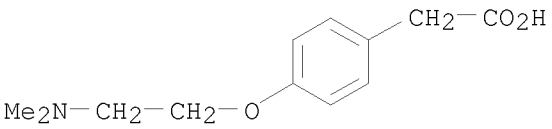
L10 ANSWER 15 OF 25 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 1958:6242 CAPLUS
DOCUMENT NUMBER: 52:6242
ORIGINAL REFERENCE NO.: 52:1101a-f
TITLE: Synthetic curare compounds. IX. Ether-esters of choline with p-hydroxyphenyl-substituted carboxylic acids
AUTHOR(S): Rosnati, Vittorio; Puschner, Heinz
CORPORATE SOURCE: Ist. super. Sanita, Rome
SOURCE: Gazzetta Chimica Italiana (1957), 87, 586-96
CODEN: GCITA9; ISSN: 0016-5603
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
AB cf. C.A. 47, 7437c. To 27.5 g. NaOH in 360 cc. H2O is added 54 g. p-HOC6H4CH2CO2H (I), 250 g. (BrCH2)2, and 750 cc. 95% EtOH, the mixture refluxed 1 hr., 3 g. NaOH added, refluxing continued 1 hr., the solvent stripped in vacuo, the residue dissolved in 300 cc. H2O and 100 cc. EtOH, and acidified with diluted H2SO4, yielding 50 g. crude product consisting mainly of p-(2-bromoethoxyphenyl)acetic acid (II), m. 108-10° (Me ester, b0.2 128-9°), and some glycol diether of I (III), m. 249-50°; di Me ester, m. 128-9°. III is separated from II by the insoly. of the diester in MeOH. II (15 g.) and 150 cc. 20% aqueous NHMe2 is heated to 110° 5 hrs., evaporated to dryness, dissolved in 200 cc. absolute EtOH, saturated with HCl gas, and refluxed 4 hrs., the product evaporated,

the residue dissolved in 30 cc. H2O, filtered, washed with Et2O, made alkaline, and repeatedly extracted with Et2O, and the exts. dried and distilled giving 8 g. Et p-(2-diethylaminoethoxyphenyl)acetate (IV), b0.2 130°; picrate, m. 116-18°. (An alternate method of preparation of IV is the condensation of I Et ester with 1-dimethylamino-2-chloroethane.) IV (8 g.) is added to 0.05 g. Na in 40 cc. 2-dimethylaminoethanol (V) and the mixture slowly distilled 1 hr. through an efficient column, 20 cc. V and 0.05 g. Na added, the distillation resumed, and the distillates stripped of V, dissolved in Et2O, washed with H2O, and fractionated, yielding 6 g. 2-dimethylaminoethyl ester of p-(2-dimethylaminoethoxy)phenylacetic acid, b0.05 130°; bisiodomethylate (VI), m. 146-8°. The phenylpropionic acid derivs. were prepared analogously, giving 3-(p-2-bromomethoxyphenyl)propionic acid, m. 131-2° (Me ester, m. 53-4°); glycol ether of 3-(p-hydroxyphenyl)propionic acid, m. 233-4° (Me ester, m. 166-7°); 3-(p-2-dimethylaminoethoxyphenyl)propionic acid (VII), m. 140-1° (Et ester, b0.1 146°); 2-dimethylaminoethyl ester of VII, b0.06 134-5° [bisiodomethylate (VIII), m. 165-6°]. According to an alternate route of synthesis, p-hydroxycinnamic acid is hydrogenated to the p-glycol monoether of phenylpropionic acid, m. 109-11°, converted to 3-(p-2-chloroethoxyphenyl)propionic acid (IX), m. 123-4°, and subsequently to 2-dimethylaminoethyl ester of IX, b0.05 175-80°. The curarelike activity of VI and VIII is strong and of short duration.

IT 857170-02-6, Acetic acid, [p-(2-dimethylaminoethoxy)phenyl]-
(derivs.)

RN 857170-02-6 CAPLUS

CN Benzeneacetic acid, 4-[2-(dimethylamino)ethoxy]- (CA INDEX NAME)



L10 ANSWER 16 OF 25 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1957:71492 CAPLUS

DOCUMENT NUMBER: 51:71492

ORIGINAL REFERENCE NO.: 51:12915b-i,12916a-i,12917a

TITLE: Derivatives of 4-amino-2-hydroxybenzoic acid. V. Basic ethers

AUTHOR(S): Clinton, R. O.; Laskowski, S. C.; Salvador, U. J.; Carroll, Patricia M.

CORPORATE SOURCE: Sterling-Winthrop Research Inst., Rensselaer, NY

SOURCE: Journal of the American Chemical Society (1957), 79, 2290-5
CODEN: JACSAT; ISSN: 0002-7863

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

OTHER SOURCE(S): CASREACT 51:71492

AB 2,4-HO(O2N)C6H3CO2Me (39.4 g.) in 1400 cc. dry PhMe treated with 4.6 g. Na and 500 cc. absolute MeOH, the MeOH distilled with stirring up to 110°,

the residual suspension refluxed 20 hrs. with stirring with 29.8 g. Et2N(CH2)2Cl in 500 cc. dry PhMe, cooled, and filtered, the filter residue washed with dry C6H6, the combined filtrate and washing evaporated in vacuo, and the oily residue treated in EtOAc with excess dry HCl in Et2O yielded 85% 2,4-Et2N(CH2)2O(O2N)C6H3CO2Me.HCl. m. 156.9-9.2°; picrate, m. 149.8-50.6° (all m.ps. are corrected). 2,4-HO(O2N)C6H3CO2Pr (24.7 g.), 16.3 g. Et2N(CH2)2Cl, and 250 cc. PrOH refluxed 8 hrs. with stirring gave after the usual procedure 1.0 g. 2,4-Et2N(CH2)2O(O2N)C6H3CO2Pr.HCl, m. 153.4-5.4°; picrate, m. 98.8-100.6°. 2,4-HO(O2N)C6H3CO2Et treated in the usual manner with p-MeC6H4SO3(CH2)2Cl gave 2,4-ClCH2CH2O(O2N)C6H3CO2Et, pale yellow platelets, 56.6-7.2°, which refluxed with a secondary amine in EtOH with NaI yielded 50-65% dialkylamino derivative The appropriate alkyl 2-hydroxy-4-nitrobenzoate, Na alkoxide, and dialkylaminoalkyl chloride under anhydrous conditions gave by the general procedure described previously (C.A. 48, 5852h) the corresponding 2,4-R2N(CH2)nO(O2N)C6H3CO2R'.HCl (I); in runs with Et2N(CH2)2Cl using the appropriate alc. as the reaction medium were obtained the following I with R = Et in the yields indicated (R' given): Me in MeOH, 5; Et in EtOH, 71; Pr in PrOH, 88; Bu in BuOH, 86; Et in EtOH from Me ester, 70. By the methods described were prepared the following I (R2N, R', n, m.p., and m.p. of picrate given): Me2N, Et, 2, 202.2-2.6°, 139.4-40.4°; Et2N, Et, 2, 143.9-4.8°, 137.8-9.0°; Et2N, Bu, 2, 117.6-18.6°, 120.5-1.6°; Et2N, Et, 3, 164.8-5.6°, 98.6-9.2°; iso-Pr2N, Et, 2, 169.1-70.7°, 160.3-3.2° (base, m. 42.0-8.9°); morpholino, Me, 2, 206.0-6.4°, 161.6-2.2°; morpholino, Et, 2, 207.0-8.0°, 154.8-5.6°; morpholino, Et, 3, 142.0-4.6°, 133.4-4.2°; 1-piperidyl, Et, 2, 191.0-1.5°, 141.7-2.9°; 1-piperidyl, Et, 3, 160.4-1.6°, 139.6-140.4°; 2-methyl-1-piperidyl, Et, 2, 180.8-2.6°, 138.0-9.0°; 2-methyl-1-piperidyl, Et, 3, 158.2-9.6°, 104.6-8.8°; 2,6-dimethyl-1-piperidyl, Et, 2, 153.0-4.0°, 207.6-9.0°. The appropriate I in EtOAc treated under anhydrous conditions with 3 moles MeI or MeBr, kept 3-20 hrs. at room temperature, and filtered gave the corresponding quaternary salt; the I in MeCN refluxed 36-72 hrs. with 3 moles of the appropriate alkyl bromide gave the corresponding salt. In this manner were prepared the following 2,5-EtO2C(O2N)C6H3O(CH2)2NMe2.RBr (R, and m.p. given): Me, - (iodide, m. 190.2-1.2°); Et, - (iodide, m. 119.1-20.2°); iso-Pr, 180.1-2.4°; iso-Bu, 137.4-8.2°; iso-Am, 150.6-3.0°; HOCH2CH2, 129.7-38.0°; PhCH2, 153.3-5.1°; 2-cyclohexylethyl, 121.9-3.5°. [2,5-EtO2C(O2N)C6H3O(CH2)2NMe2]2.(CH2)nBr2 (n and m.p. given): 2, 164.1-72.0°; 3, 185.1-92.0°; 4, 179.0-86.9°; 5, 184-7° (decomposition) with sintering from 152° when immersed at 25°; 6, 192.3-5.9°. 2,5-RO2C(O2N)C6H3O(CH2)2NEt2.MeI (R, and m.p. given): Me, 162.5-3.0°; Et, 143.1-4.6° (bromide, m. 150.6-1.6°); Pr, 143.2-4.6°; Bu, 118.2-20.3°. [2,5-EtO2C(O2N)C6H3O(CH2)2NEt2]2.(CH2)nBr2, (n and m.p. given): 2, 146.7-8.7°; 4, 143.2-6.8°; 6, 150.7-8.2°. 2,5-R'O2C(O2N)C6H3O(CH2)nNR2.R''X (R2N, R', R'', n, X, and m.p. given): Et2N, Et, Et, I, 2, 140.7-1.9°; Et2N, Et, Me, I, 3, 149.0-9.6°; iso-Pr2N, Et, Me, I, 2, 183.7-4.2°; morpholino, Me, Me, I, 2, 209.0-11.0°; morpholino, Et, Me, I, 2, 190.5-1.3°; morpholino, Et, Me, I, 3, 161.1-1.7°; 1-piperidyl, Et, Me, I, 2, 147.7-8.9°; 1-piperidyl, Et, Me, I, 3,

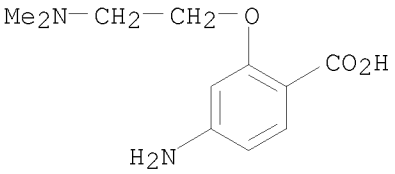
166.9-7.9°; 2-methyl-1-piperidyl, Et, Me, I, 2, 159.8-61.0°; 2-methyl-1-piperidyl, Et, Me, I, 3, 165.5-6.5°; 2,6-dimethyl-1-piperidyl, Et, Me, I, 2, 192.3-2.9°. The appropriate alkyl 2-(dialkylaminoalkoxy)-4-nitrobenzoate (0.01 mole) and 0.02 mole 2,4-HO(O2N)C6H3CN in EtOAc yielded essentially quantitatively the corresponding alkyl 2-(dialkylaminoalkoxy)-4-nitrobenzoate 2-cyano-5-nitrophenolate (alkyl, dialkylaminoalkoxy group, crystal form, and m.p. given): Et, Et2N(CH2)2O, canary-yellow prisms, 76.0-8.0°; Et, 3-piperidinopropoxy, short blunt orange needles, 125.2-6.0°; Et, 3-morpholinopropoxy, hair-like yellow-orange needles, 137.2-8.3°. 2,4-Et2N(CH2)2O(O2N)C6H3CO2Et.HCl (15.0 g.), 18.3 g. Na2CO3, and 200 cc. 50% EtOH refluxed 4 hrs. with stirring, the EtOH removed in vacuo, the aqueous residue acidified with concentrated HCl to Congo red and saturated with (NH4)2SO4, and the precipitate filtered off yielded 13.8 g. 2,4-Et2N(CH2)2O(O2N)C6H3CO2H (II) HCl salt, m. 212.5-13.9° (from MeOH). II.HCl (31.9 g.), 8.4 g. NaHCO3, and 500 cc. absolute EtOH refluxed 3 hrs. with stirring, cooled, filtered, and evaporated in vacuo gave 25.0 g. II, m. 164.6-6.6°; picrate, cottony yellow needles, m. 179.2-80.4°. Similarly was prepared 2,4-Me2N(CH2)2O(O2N)C6H3CO2H, cream-colored plates, m. 193.1-4.1° (from absolute EtOH) [HCl salt, pale yellow needles, m. 208.0-9.6° (from absolute EtOH); picrate, clusters of yellow needles, m. 181.8-2.6°], and 2-(3-piperidinopropoxy)-4-nitrobenzoic acid HCl salt, pale yellow cotton needles, m. 216.8-17.5° (from absolute EtOH) [picrate, canary-yellow needles, m. 143.0-5.0° (from absolute EtOH)]. The appropriate alkyl 2-(dialkylaminoalkoxy)-4-nitrobenzoate base or HCl salt reduced in the appropriate dilute alc. with Fe and HCl or catalytically at 25° in the appropriate alc. over PtO2 gave the corresponding 4,2-H2N[R2N(CH2)nO]C6H3CO2R' (R2N, R', m.p. of phosphate, and m.p. of picrate given). With n = 2: Me2N, Et, 176.3-7.3°, 140.2-1.2° (base, m. 94.2-5.6°); Et2N, Me, 195.8-6.8°, 119.0-20.4° (dipicrate); Et2N, Et, 168.7-9.6°, 131.6-3.2° (di-HCl salt, m. 173.6-3.9°); Et2N, Pr, 153.0-4.0°, 140.4-1.2°; Et2N, Bu, 154.5-5.5°, 120.8-2.6°; iso-Pr2N, Et, 186.0-7.0°, -(flavianate, m. 196.8-7.8°); morpholino, Me, 151.3-2.1° (diphosphate), 168.5-9.7°; morpholino, Et, 196.3-6.9°, 165.8-6.8° (base, m. 98.0-9.8°); piperidino, Et, 220.8-1.4°, 159.0-60.0° (base, m. 107.3-8.5°); 2-methylpiperidino, Et, -, 172.4-3.6° (base, m. 91.2-2.4°); 2,6-dimethylpiperidino, Et, 211.0-11.8°, 188.8-9.6°. With n = 3: Et2N, Et, 151.5-3.2°, 146.2-7.0°; morpholino, Et, 143.3-4.4°, 210.4-11.4° (base, m. 106.8-8.0°); piperidino, Et, 160.2-1.6°, 218.0-18.7° (base, 109.2-10.1°); 2-methylpiperidino, Et, 136.4-8.3°, 180.8-3.0° (base, m. 112.4-13.8°). The appropriate alkyl 2-(dialkylaminoalkoxy)-4-nitrobenzoate quaternary and bisquaternary salts gave similarly by Fe-HCl or catalytic reduction the 4-NH2 analogs. In this manner were prepared 5,2-H2N(R'O2C)C6H3O(CH2)nNR2.R''X (R2N, R', R'', X, and m.p. given). With n = 2: Me2N, Et, Me, I, 204.2-5.2°; Me2N, Et, Et, I, 172.3-5.3°; Me2N, Et, iso-Pr, Br, 190.0-2.2°; Me2N, Et, HOCH2CH2, Br, 138.9-42.3°; Me2N, Et, 2-cyclohexylethyl, Br, 101.6-5.1°; Me2N, Et, (CH2)2, Br, 190.0-95° (decomposition); Me2N, Et, (CH2)4, Br, 150° (indefinite above 160° with decomposition); Me2N, Et, (CH2)5, Br, 125° (indefinite above 190° with decomposition);

Me2N, Et, (CH2)6, Br, 200.7-2.5°; Et2N, Me, Me, I, 127.4-9.0°; Et2N, Et, Me, Br, 160.3-2.1°; Et2N, Et, Me, I, 139.2-41.1°; Et2N, Pr, Me, I, 127.4-9.6°; Et2N, Bu, Me, I, 88.2-92.4°; Et2N, Et, Et, I, 141.2-3.8°; morpholino, Et, Me, I, 182.7-3.7°; piperidino, Et, Me, I, 167.4-8.4°; 2,6-dimethylpiperidino, Et, Me, I, 123.4-6.4°. With n = 3: Et2N, Et, Me, I, 125.0-6.0°; morpholino, Br, Me, I, 151.9-3.1°; piperidino, Et, Me, I, 150.1-50.6°. The appropriate 2-(dialkylaminoalkoxy)-4-nitrobenzoic acids or their HCl salts reduced catalytically yielded the corresponding 4-amino-2-(2-dialkylaminoalkoxy)benzoic acids (dialkylaminoalkoxy group, crystal form, arid m.p. given): Et2N(CH2)2O, needles, 158.0-8.8° (decomposition) [picrate, canary-yellow needles, m. 187.5-8.3° (from EtOH)]; Me2N(CH2)2O, -, -(HCl salt, needles, m. 145.5-7.2° with decomposition); 3-piperidinopropoxy, -, -(HCl salt, tan needles, m. 162.1-2.8° with decomposition). Reductive alkylation of the appropriate 4-NH2 bases with an aldehyde, Zn dust, and AcOH gave 4,2-BuNH(Et2NCH2CH2O)C6H3CO2Et.HCl, cream-colored needles, m. 160.5-1.8° (from absolute EtOH-EtOAc) [flavianate, yellow-orange plates, m. 164.6-5.6° (from EtOH)], 4,2-HO(CH2)5NH(Et2NCH2CH2O)C6H3CO2Et.HCl, cottony needles, m. 132.2-3.4° (from absolute EtOH hexane) (flavianate, cottony orange needles, m. 126.0-6.4°), Et 4-(2,2-dimethyl-3-hydroxypropylamino)-2-[2-(2,6-dimethylpiperidino)ethoxy] benzoate, needles, m. 90.0-1.0° (from C6H6).

IT 807293-69-2, Benzoic acid, 4-amino-2-(2-dimethylaminoethoxy)-
856788-92-6, Benzoic acid, 4-amino-2-(2-diisopropylaminoethoxy)-
(derivs.)

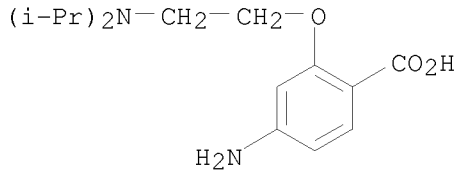
RN 807293-69-2 CAPLUS

CN Benzoic acid, 4-amino-2-[2-(dimethylamino)ethoxy]- (CA INDEX NAME)



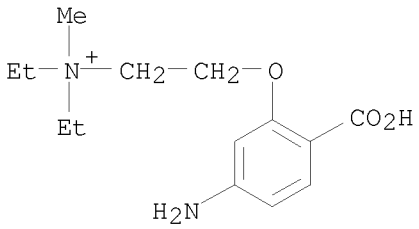
RN 856788-92-6 CAPLUS

CN Benzoic acid, 4-amino-2-[2-[bis(1-methylethyl)amino]ethoxy]- (CA INDEX NAME)

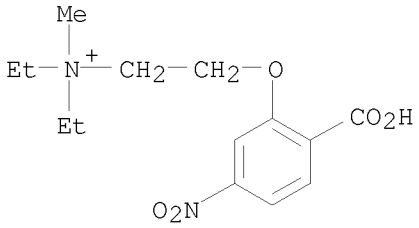


IT 857174-71-1, Ammonium, [2-(5-amino-2-carboxyphenoxy)ethyl]diethylmethyl- 857179-13-6, Ammonium, [2-(2-carboxy-5-nitrophenoxy)ethyl]diethylmethyl-
(halides, esters)

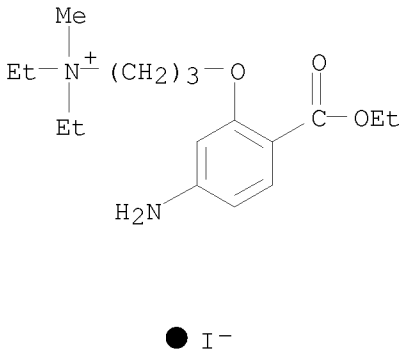
RN 857174-71-1 CAPLUS
CN Ethanaminium, 2-(5-amino-2-carboxyphenoxy)-N,N-diethyl-N-methyl- (CA INDEX NAME)



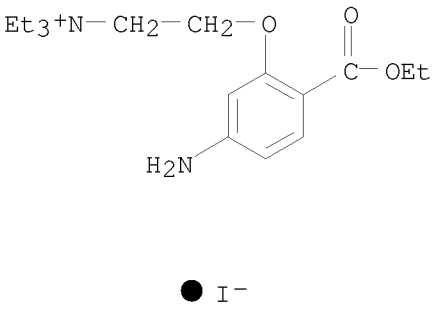
RN 857179-13-6 CAPLUS
CN Ethanaminium, 2-(2-carboxy-5-nitrophenoxy)-N,N-diethyl-N-methyl- (CA INDEX NAME)



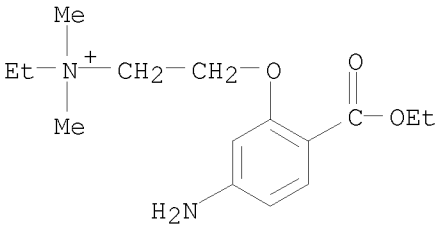
IT 857174-47-1P, Ammonium, [3-(5-amino-2-carboxyphenoxy)propyl]diethylmethyl-, iodide, Et ester
857174-56-2P, Ammonium, [2-(5-amino-2-carboxyphenoxy)ethyl]triethyl-, iodide, Et ester 857174-64-2P, Ammonium, [2-(5-amino-2-carboxyphenoxy)ethyl]ethyldimethyl-, iodide, Et ester
RL: PREP (Preparation)
(preparation of)
RN 857174-47-1 CAPLUS
CN 1-Propanaminium, 3-[5-amino-2-(ethoxycarbonyl)phenoxy]-N,N-diethyl-N-methyl-, iodide (1:1) (CA INDEX NAME)



RN 857174-56-2 CAPLUS
CN Ethanaminium, 2-[5-amino-2-(ethoxycarbonyl)phenoxy]-N,N,N-triethyl-, iodide (1:1) (CA INDEX NAME)



RN 857174-64-2 CAPLUS
CN Ethanaminium, 2-[5-amino-2-(ethoxycarbonyl)phenoxy]-N-ethyl-N,N-dimethyl-, iodide (1:1) (CA INDEX NAME)



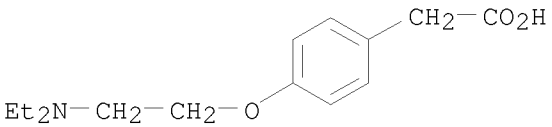
L10 ANSWER 17 OF 25 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 1956:69464 CAPLUS
DOCUMENT NUMBER: 50:69464
ORIGINAL REFERENCE NO.: 50:13044d-h
TITLE: Aryl ketones and thio morpholides in the synthesis of 8-substituted xanthines
AUTHOR(S): Hager, Geo. P.; Kramer, Stanley P.
CORPORATE SOURCE: Univ. of Maryland, Baltimore
SOURCE: Journal of the American Pharmaceutical Association (1912-1977) (1955), 44, 649-53
CODEN: JPHAA3; ISSN: 0003-0465
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
AB The following 8-substituted benzyltheophyllines were prepared by heating equimolar amts. of the appropriate phenylacetic acid and 1,3-dimethyl-5,6-diaminouracil (I) just above the m.p. until the mixture resolidified, dissolving the product in boiling 5% NaOH, precipitating with CO2,

and recrystg. from HOAc, absolute EtOH, HCONMe2, or mixts. of HOAc with H2O, EtOH or Et2O. The following compds. were prepared, 8-benzyl substituent and m.p. given: m-HO, above 300°; p-HO, above 300°; 3,4-(HO)2, above 300°; m-MeO, 251-2°; p-MeO, 276.7-7.5°; p-EtO, 256°; p-PhCH2O, 235.5-57°; p-Et2NCH2CH2O, 189.5-90°; 3,4-(MeO)2, 246-7°; 3,4-CH2O2, above 300°; α-MeO, 193.5-4°. 8-Benzyltheophylline, m. 297-8°, was prepared in 32% yield by heating I and phenylthioacetomorpholide 7 hrs. at 110-75°. 4-Aminophenylthioacetomorpholide gave 5% 8-(4-aminobenzyl)theophylline, m. 297-8°. From 7.2 g. PhAc, 2.4 g. S, and 5.1 g. I refluxed 30 min. at 155-70° and 6 hrs. at 170° and worked up as above was obtained 30% 8-benzyltheophylline, m. 276-8°. Substitution of styrene or trithioacetophenone for PhAc in the above reaction gave little or no product. Ethylenediamine-p-MeC6H4SO3H, S, and PhAc in 10 hrs. at 170-85° gave after treatment with HCl in absolute EtOH 3.7% of "2-benzyl-2-imidazolinium chloride," m. 171-3°. p-HOC6H4CH2CO2Et (22.5 g.), 44 g. Et2NCH2CH2Cl.HCl, 138 g. K2CO3, and 754 ml. dry Me2CO refluxed 14 hrs. gave 20 g. p-Et2NCH2CH2OC6H4CH2CO2Et (II), b2 155-64°; HCl salt, m. 131.5-2.5°. II (6 g.), 5 ml. HCl, and 40 ml. H2O refluxed 8 hrs., evaporated and the residue recrystd. from Me2CO gave 5.5 g. p-Et2NCH2CH2OC6H4CH2CO2H.HCl, m. 127-8.5°. p-HOC6H4CH2CO2H (15.2 g.) added to 13.6 g. NaOEt in 75 ml. absolute EtOH, the solvent removed in vacuo and the residue refluxed 5 hrs. with 125 ml. HCONMe2 and 67.8 g. Et2NCH2CH2Cl gave 10% p-Et2NCH2CH2OC6H4CH2CO2CH2CH2NEt2, b2 190-211°; di-HCl salt, m. 158-9°.

IT 802559-45-1, Acetic acid, [p-(2-diethylaminoethoxy)phenyl]-
(derivs.)

RN 802559-45-1 CAPLUS

CN Benzeneacetic acid, 4-[2-(diethylamino)ethoxy]- (CA INDEX NAME)



L10 ANSWER 18 OF 25 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1956:48568 CAPLUS

DOCUMENT NUMBER: 50:48568

ORIGINAL REFERENCE NO.: 50:9321c-i

TITLE: The o-Claisen transfer. Experiments with carbon-14. VII. Also Claisen rearrangements. V. The ortho-Claisen rearrangement

AUTHOR(S): Fahrni, P.; Haegele, W.; Schmid, K.; Schmid, H.

CORPORATE SOURCE: Univ. Zurich, Switz.

SOURCE: Helvetica Chimica Acta (1955), 38, 783-9
CODEN: HCACAV; ISSN: 0018-019X

DOCUMENT TYPE: Journal

LANGUAGE: German

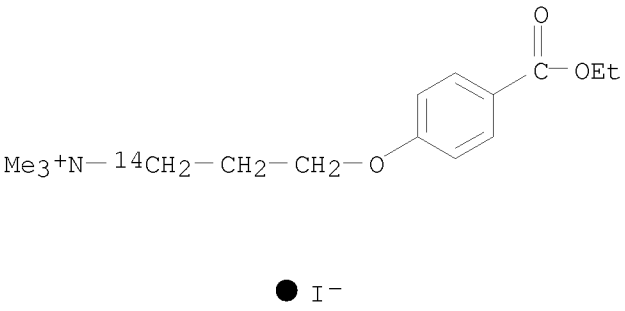
AB The ortho-Claisen rearrangement of 2,4-disubstituted Ph allyl ethers (I), contrary to that of 2-monosubstituted I (II), is uniform. II normally form the 6-allyl-2-substituted phenols but also 4-allyl-2-substituted

phenols. The exact reaction was proven with 4,2-Me(CH2:CHC14CH2)C6H3O:CH2CH:CH2 (III) and 4,2-MeO2C(CH2:CHC14CH2)C6H3OCH2:CHCH2 (IV). It was investigated if the intermediates in the transfer of III were 2,2-diallyl-4-methyl-3,4-cyclohexadien-1-one and 2,4-diallyl-4-methyl-2,5-cyclohexadien-1-one. A 2,6-diallylphenol free of C14H2:CHCH2 was obtained. ClCH2CH2C14H2OH (7.54 g.) and 14.65 g. 4-HOC6H4CO2Et refluxed 100 hrs. in 40 cc. Me2CO with 26.5 g. pulverized KI and 13.2 g. K2CO3, the cooled mixture treated with H2O, extracted with Et2O, and the extract washed with H2O, 2% NaOH, and brine, dried, and evaporated yielded 12.55 g. (70%) p-EtO2CC6H4OCH2CH2C14H2OH (V) m. 40.5-1.5° (from Et2OC5H12). The purest SOCl2 (6.78 g.) in 13 cc. CHCl3 added dropwise to 8.483 g. V in 26 cc. CHCl3 and 3.2999 g. pyridine, the mixture kept 2 hrs. in the dark, then boiled 45 min., and the Cl compound separated in the usual manner, converted into the iodo compound with NaI in Me2CO, and finally treated with a 4-fold amount of NMe3 in alc. yielded 11.9 g. p-EtO2CC6H4OCH2CH2C14H2NMe3I, m. 172.5-4°; 22.03 g. of this compound stirred 48 hrs. in a vibro-mixer with 30 g. AgNO3 in H2O, the mixture filtered, the filtrate evaporated to 50° in vacuo, the crystalline residue heated 16 hrs. to 110-20° with 240 cc. 33% NaOH, 50 cc. H2O added, the mixture heated 10 hrs. to 110-20°, cooled, acidified with 1:1 HCl, left overnight, filtered through glass wool, and the filter and filter cake extracted with Et2O in a Soxhlet yielded 7.67 g. 4-C14H2:CHCH2OC6H4CO2H, m. 158-60° (from alc.); its Me ester (made with N2CH2), (4.606 g.) heated 20 hrs. with 9 cc. Et2NPh under a high vacuum in a boiling BzMe bath, the product dissolved in Et2O, and the extract washed and distilled (b0.05 80-100°) gave 3.45 g. 2,4-C14H2:CHCH2(MeO2C)C6H3OH, (VI), m. 92-3° (from CCl4 and Et2O-C5H12), which with MeI and K2CO3 in Me2CO yielded 2,4-C14H2:CHCH2(MeO2C)C6H3OMe (VII), b0.05 125-35°, colorless oil. VII treated in known manner with OsO4 in pyridine gave 2,4-C14H2(OH)CH(OH)CH2(MeO2C)C6H3OMe, m. 173-5° (from AcOEt). VI (2.384 g.) in 9.5 cc. MeOH, and 0.28 g. Na treated with 1.67 g. CH2:CHCH2Br dropwise within 10 min. at 95-105°, heated 2 hrs., and worked up as usual gave 2.715 g. IV, colorless oil, b0.04 110-20°; free acid, m. 140.5-1.0°. IV (2.197 g.) and 4 cc. Me2NPh heated 24 hrs. to 200° in a high vacuum and distilled yielded 1.5 g. 4,2,6-MeO2C(CH2:CHC14H2)2C6H2OH, m. 58-9.5° (from C5H12-C6H6); Me ether, colorless oil, b0.01 105-15°. The corresponding compds., III, b10 110-20°, and 4,2,6-Me(CH2:CHC14H2)2C6H2OMe, b0.05 70-80°, were prepared similarly.

IT 855945-29-8P, Ammonium, [3-(p-carboxyphenoxy)propyl-1-C14]trimethyl-, iodide, Et ester
RL: PREP (Preparation)
(preparation of)

RN 855945-29-8 CAPLUS

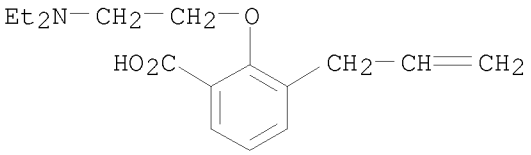
CN Ammonium, [3-(p-carboxyphenoxy)propyl-1-C14]trimethyl-, iodide, Et ester (5CI) (CA INDEX NAME)



L10 ANSWER 19 OF 25 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 1951:46932 CAPLUS
DOCUMENT NUMBER: 45:46932
ORIGINAL REFERENCE NO.: 45:7976h-i,7977a-b
TITLE: Syntheses of basic phenol alkyl ethers. X. Derivatives of isoeugenol, resorcinol, and salicylic acid
AUTHOR(S): Senda, Shigeo
CORPORATE SOURCE: Univ. Kyoto
SOURCE: Yakugaku Zasshi (1950), 70, 561-4
CODEN: YKKZAJ; ISSN: 0031-6903
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

AB cf. preceding abstract The Na salt of isoeugenol and Cl(CH2)2NEt2 (I) give 2,4-MeO(MeCH:CH)C6H3O(CH2)2NEt2 (II), b4 185-7°. Isoeugenol, K2CO3, and C3H5Br in Me2CO give 2,4-MeO(MeCH:CH)C6H3OC3H5 (III), b4 153°. Heating III at 280-90° in vacuo gives 2,4,6-MeO(MeCH:CH)(C3H5)C6H2OH (IV), b3 145°. Adding 5.5 g. IV to 0.68 g. Na in 25 ml. MeOH, then 6 g. I, heating at 100° 5 hrs., and distilling gives 1 g. 2,4,6-MeO(MeCH:CH)(C3H5)C6H2O(CH2)2NEt2 (V), b5 185-8°. Allyl transition by heating 22 g. m-(C3H5O)2C6H4 in vacuo 40 min. at 260-80° gives 9 g. 4,6,1,3-(H5C3)2C6H2(OH)2 (VI), b1 146-7°. Heating 9 g. VI, 2.2 g. Na in 40 ml. MeOH, and 12 g. I on a water bath 7 hrs. and treating as in II gives 5.5 g. 4,6,1,3-(C3H5)2C6H2(OCH2CH2NEt2)2 (VII), b3 199°. Heating 10 g. 2,3-HO(C3H5)C6H3CO2Me, 1.2 g. Na in 30 ml. MeOH, and 7 g. I 6 hrs. at 100°, removing the MeOH, acidifying with HCl, taking up with AcOEt, and shaking up with aqueous NaOH gives 6.5 g. 2,6-C3H5(MeO2C)C6H3O(CH2)2NEt2 (VIII), b4 160°; 6-EtO2C analog, b8 183-5°. Heating 25 g. salicylic acid in 60 ml. acetone with 70 g. K2CO3 and 50 g. C3H5Br at 100° 8 hrs. and treating as in II gives 3.5 g. 2,6-H5C3(H5C3O2C)C6H3O(CH2)2NEt2 (IX), b3 165°; 2,3-HO(C3H5)C6H3CO2CH2CH2NEt2, b3 175°. VIII showed on the uterus of the guinea pig in vivo a contracting action stronger than that of Gravitol (I. G.) and about the same toxicity on the mouse.

IT 860692-96-2, Benzoic acid, 3-allyl-2-(2-diethylaminoethoxy)-, (esters)
RN 860692-96-2 CAPLUS
CN Benzoic acid, 2-[2-(diethylamino)ethoxy]-3-(2-propen-1-yl)- (CA INDEX NAME)



L10 ANSWER 20 OF 25 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 1949:36533 CAPLUS
DOCUMENT NUMBER: 43:36533
ORIGINAL REFERENCE NO.: 43:6590d-i,6591a-g,6592a-f
TITLE: Synthetic curare compounds. II. Aryl aliphatic derivatives with double quaternary ammonium function
AUTHOR(S): Fusco, Raffaello; Chiavarelli, Stefano; Palazzo, Giuseppe; Bovet, Daniel
SOURCE: Gazzetta Chimica Italiana (1948), 78, 951-64
CODEN: GCITA9; ISSN: 0016-5603
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

AB cf. C.A. 43, 2190d. The purpose was (1) to ascertain the influence on the pharmacodynamic properties of the O bridge which is present in aromatic polyesters and phenolic polyethers and in natural compds. of the tubocurarine group, and (2) to compare the properties of synthetic curare derivs. containing aromatic rings already studied with those of the aliphatic type described by Barlow and Ing (C.A. 42, 6930b), Paton and Zaimis (C.A. 42, 6930d), and Glock, et al. (C.A. 43, 6737b). No previous study has been reported of the pharmacol. properties of aryl aliphatic derivs. with double quaternary ammonium structure. A study of the various methods for preparing p-C6H4(CH2Cl)2 (I) led to the development of the following method as best. PhCH2Cl (1265 g.), 300 g. trioxymethylene, and 1360 g. anhydrous ZnCl2, saturated at 30-40° with HCl, heated at 60° until the exothermic reaction is complete (about 45 min.), then 10 min. at 80°, treated with water, and the C6H6 layer diluted with warm C6H6, washed again with water, distilled to a small volume, and fractionated in vacuo, yields about 400 g. PhCH2Cl; the residue allowed to crystallize ice-cold, and the product (275 g.) washed with petr. ether, and purified by C6H6, EtOH, or ligroin, yields I. I (9 g.) and HNMe2 (approx. 4 mols.) in 100 cc. C6H6 heated in a sealed tube at 60° overnight, taken up in water, NaOH added, and the C6H6 layer dried with NaOH, and distilled in vacuo, yield 7.5 g. (75%) N,N,N',N'-tetramethyl-α,α'-p-xylenediamine, p-C6H4(CH2NMe2)2 (II), b1 102°. I (17.5 g.) and 30 g. HNEt2, refluxed 3 hrs. (until, when diluted with acidified water, the mixture is clear), taken up in water, K2CO3 added, extracted with Et2O, and the extract dried with K2CO3 and distilled in vacuo, yield 17.5 g. (70%) N,N,N',N'-tetraethyl-α, α'-p-xylenediamine (III), b1 110°. Excess MeI added cautiously to II in Me2CO (heat is evolved), and refluxed briefly, yields almost 100% p-xylylenebis-[trimethylammonium iodide] (IV), sinters 286°, m. 298-300° (decomposition). Similarly EtI and II in Me2CO, refluxed 2 hrs., yield almost 100% of p-xylylenebis[ethyldimethylammonium iodide] (V), m. 240-1° (decomposition). MeI and III in Me2CO, refluxed 2 hrs., give, after purification by dilute EtOH, a high yield of p-xylylenebis[diethylmethylammonium iodide] (VI), m. 228-30° (decomposition). It was found impossible to make I react with NEt3; but 8.8 g. I in 70 cc. anhydrous EtOH and 16.7 g. NaI in 40 cc. anhydrous EtOH, refluxed 30 min., taken up in water, filtered,

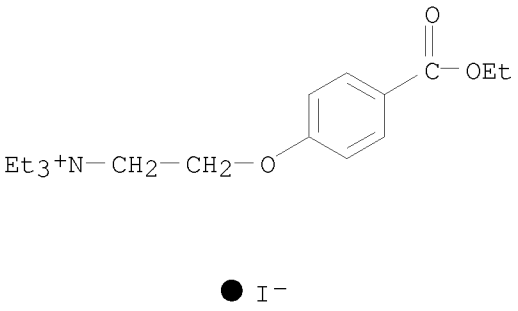
dried in vacuo, and purified by EtOH, yield 13.5 g. p-C6H4(CH2I)2 (VII), m. 166-9° (cf. Finkelstein, C.A. 4, 2441). VII and 2 mols. NEt3, heated in a sealed tube 30 min. at 80°, taken up in EtOH, water added, clarified by animal charcoal, excess NaOH added, and the precipitate purified by EtOH, yield p-xylylenebis[triethylammonium iodide] (VIII), m. 221-2° (decomposition). A very high yield is obtained when it is prepared from III and EtI in Me2CO by the foregoing technique. The method of Ruggli, et al. (C.A. 30, 1759.1) for preparing p-C6H4(CH2CH2NH2)2 (IX), b4 137-8°, was modified by heating p-C6H4(CH2CN)2, H, Raney Ni, and alc. NH3 15 min. at 90° under 90 atmospheric pressure. IX (2.8 g.) in MeOH, 6 g. KOH in 50 cc. MeOH, and 22 g. MeI, refluxed 1 hr., evaporated, the residue dissolved in hot water, filtered, allowed to stand, and the precipitate washed with MeOH and purified by water, yield 1,4-bis(2-dimethylaminoethyl)benzene-2-MeI (X), m. 314° (decomposition). Similarly 1.6 g. IX and EtI yield 3.1 g. of 1,4-bis(2-diethylaminoethyl)benzene-2EtI (XI), m. 262-3°. IX, KOH, and PrI in PrOH, refluxed 2 hrs., and the product purified by PrOH, yield 1,4-bis(2-dipropylaminoethyl)benzene-2PrI (XII), m. 214-15° (decomposition). 2,4,1,5-Me2C6H2(CH2Cl)2 (20.5 g.) in 120 cc. MeOH and aqueous NaCN (12.5 g. in 37 cc.), refluxed 30 min., 300 cc. water added, made ice-cold, and the precipitate purified by MeOH and animal charcoal, yield 10 g. of 1,5-dimethyl-2,4-bis(cyanomethyl)benzene (XIII), m. 88-9°. XIII (10 g.) in 150-200 cc. anhydrous EtOH, saturated at 0° with NH3, hydrogenated with 1-2 g. Raney Ni at 80-6° and 100 atmospheric pressure (about 1.5 hrs.), and the filtered mixture distilled in vacuo, yields 7 g. of 1,5-dimethyl-2,4-bis(2-aminoethyl)benzene (XIV), b2 147°. Following the procedure used in the preparation of XI, 1.9 g. XIV yields 5.5 g. 1,5-dimethyl-2,4-bis(2-diethylaminoethyl)benzene-2EtI (XV), m. 255-6° (decomposition). The following method for preparing 2,4-bis(chloromethyl)anisole (XVI) is an improvement over other published methods. Anisole (100 g.), 142 g. 37% HCHO, and 795 g. concentrated HCl, saturated with HCl (keeping cold by ice-salt), allowed to stand 1 hr. at 10-12°, heated 3 hrs. at 60°, the upper layer poured onto ice, the precipitate dissolved in Et2O, washed, dried by CaCl2, the Et2O distilled, the residue taken up in petr. ether, made ice-cold, and the precipitate purified by petr. ether, yields 104 g. (58%) XVI. XVI (100 g.) and NaCN (calculated weight) in anhydrous MeOH precipitate NaCl; the product, diluted, extracted with a solvent (not specified), and the extracted product fractionated in vacuo, yields in great part a distillate b2-3 120-195° and 8.5 g. of impure 2,4-bis(cyanomethyl)anisole (XVII), b2 approx. 200°. By hydrogenation, 8 g. XVII yields 2,4-bis(2-aminoethyl)anisole (XVIII), b4 164°. Ethylation of XVIII is carried out as above, except that the final product is extracted and purified by anhydrous EtOH; the product is 2,4-bis(2-diethylaminoethyl)anisole-2EtI (XIX), m. 236-7° (decomposition). p-HOC6H4CO2Et (8 g.) in alc., NaOEt (from 1.25 g. Na and 30 cc. anhydrous EtOH), and Et2NCH2CH2Cl (XX) [from 11 g. Et2NCH2CH2Cl.HCl (XXI) by treatment with K2CO3 and extraction with Et2O], heated in a sealed tube 24 hrs. at 140°, filtered, evaporated in vacuo, the residue taken up in water, K2CO3 added, extracted with Et2O, the extract evaporated, and the residue fractionally distilled in vacuo, give a small yield of Et p-(2-diethylaminoethoxy)-benzoate (XXII), b2 168-9°. With EtI,

XXII forms the ethiodide, p-IEt3NCH2CH2OC6H4CO2Et. XXII (6 g.), 5 cc. concentrated HCl, and 40 cc. water, refluxed 8 hrs., concentrated, allowed to stand, and the precipitate purified by aqueous Me2CO, yield 5.5 g. p-(2-diethylaminoethoxy)benzoic acid, m. 170-1°. p-HOC6H4CO2H (3.6 g.) in a min. of anhydrous EtOH, NaOEt (from 1.25 g. Na and 24 cc. anhydrous EtOH), and XX (from 10 g. XXI), heated in a sealed tube overnight at 130°, filtered, evaporated, the residue taken up in anhydrous Et2O, filtered, evaporated, and the residue distilled in vacuo, yield XXII. A method different from that of Rohmann and Scheurle (C.A. 30, 4160.7) was used for preparing p-HOC6H4CO2CH2CH2NEt2 (XXIII). HCl gas, passed through 13.8 g. p-HOC6H4CO2H and 11.7 g. Et2NCH2CH2OH (XXIV) at 115-20° for several hrs., taken up in 6 parts by weight of hot EtOH, allowed to cool, and the precipitate purified by EtOH, yields XXIII.HCl (XXV), m. 185-6°. XXV (6 g.) in anhydrous EtOH, EtONa (from 1.25 g. Na and 25 cc. anhydrous EtOH), and XX (from 5.5 g. XXI), heated in a sealed tube 48 hrs. at 120°, and the same procedure followed as before, yields 2.6 g. XXII. HCl gas, passed through 7.8 g. XXII and 4 g. XXIV 8 hrs. at 110-20°, taken up in water, K2CO3 added, extracted with Et2O, and the extract dried, evaporated, and distilled in vacuo, yields p-Et2NCH2CH2OC6H4CO2CH2CH2NEt2 (XXVI), b2 190-5°. With excess EtI, and purification of the product by anhydrous EtOH, it yields 2-diethylaminoethyl p-(2-diethylaminoethoxy)benzoate-2EtI, p-IEt3NCH2CH2OC6H4CO2CH2CH2NEt3I (XXVII), m. 175-6° (decomposition). The pharmacol. properties of 10 of the compds. were tested by endovenous injection in rabbits. The following data give the "head-drop" dose (cf. preceding work, loc. cit.) and lethal dose in mg./kg., resp.: IV, 25, 40; V, 15, 15; VI, 8, 15; VIII, 2, 3; X, 20, 25; XI, 3, 4; XII, 10, 12; XV, 2, 3; XIX, 2, 3; XXVII, 4, 15. These results show that, with progressive substitution of Et by Me groups, the curarizing power of any series of compds. decreases, but that neither the position of the chain carrying the ammonium ion nor the number of C atoms which sep. the N from the nucleus has any great influence on the curarizing power. The curarizing power of XXVII is, as expected, of the same magnitude as that of p-C6H4(OCH2CH2NEt3I)2 and p-C6H4(CH2CH2NEt3I)2, which are equally active. Furthermore, this activity is about the same as that of VIII; hence the presence of an oxygenated group has no significant influence on the curarizing power.

IT 857159-93-4P, Ammonium, [2-(p-carboxyphenoxy)ethyl]triethyl-, iodide, Et ester
RL: PREP (Preparation)
(preparation of)

RN 857159-93-4 CAPLUS

CN Ethanaminium, 2-[4-(ethoxycarbonyl)phenoxy]-N,N,N-triethyl-, iodide (1:1) (CA INDEX NAME)



L10 ANSWER 21 OF 25 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 1949:17410 CAPLUS
DOCUMENT NUMBER: 43:17410
ORIGINAL REFERENCE NO.: 43:3360h-i,3361a-i,3362a-g
TITLE: Biosynthesis of penicillins. V. Substituted phenylacetic acid derivatives as penicillin precursors
AUTHOR(S): Corse, Joseph W.; Jones, Reuben G.; Soper, Quentin F.; Whitehead, Calvert W.; Behrens, Otto K.
SOURCE: Journal of the American Chemical Society (1948), 70, 2837-43
CODEN: JACSAT; ISSN: 0002-7863
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

AB cf. C.A. 43, 2274b. A description is given of substituted PhCH2CO2H derivs. which have been tested as precursor substances in the preparation of new penicillins. p-HOC6H4CH2CO2Et (I) (36 g.) and 38 g. PhCH2Cl in 300 mL. absolute EtOH containing 13.3 g. MeONa, refluxed overnight and the ester refluxed overnight with 70 g. KOH in 400 mL. EtOH and 70 mL. H2O, give 15.2 g. (p-benzyloxyphenyl)acetic acid (II), m. 120-1°. I (15.2 g.) in 200 mL. H2O and 48.4 mL. 4.135 N NaOH (stirred in an ice bath), treated dropwise with 12 g. ClCO2Et, the mixture stirred 2 h., and 32 mL. 4 N HCl added, gives (p-carbethoxyoxyphenyl)acetic acid, m. 78-9°. II (15.2 g.) in 30 mL. SOCl2, the mixture kept overnight, the residue treated with 11.7 g. DL-valine and 16 mL. 12 N NaOH in 200 mL. H2O, gives N-(p-benzyloxyphenylacetyl)-DL-valine (III), m. 144-5°, S 1.37 (S is the stimulation; compds. were tested at 0.0008 M concentration; the values represent the ratio units in test container/units in control container). The following analogs of III were prepared (R in RC6H4CH2CONHCH(CHMe2)CO2H) (S is 1 unless otherwise given): o-NO2 m. 173-5°, m-NO2 m. 153-8° (S 0.88), p-NO2 m. 134-5° (S 1.49), o-NH2 m. 238-41° (S 1.37), p-NH2 m. 220-7° (prepared by catalytic reduction of the NO2 derivs.), o-Cl m. 122-4°, p-Cl m. 144-5° (S 1.33), p-CN m. 138-40° (S 1.24), p-I m. 148-50°, p-iso-Pr m. 114-15°, p-MeO m. 129° (S 1.52), 2,4,6-tri-Me m. 130-2° N-(p-nitrophenylacetyl)isoleucine m. 113-15°. The following esters were prepared by treating the substituted PhMe with Br and the resulting PhCH2Br with KCN, hydrolyzing the nitrile with aqueous alc. H2SO4, and esterifying with MeOH-H2SO4: Me (3,4-dibromophenyl)acetate m. 44-5° 3,4,5-tri-Br analog m. 78-9° 4-bromo-3-chloro analog m. 42-3°. Et (o-fluorophenyl)acetate, b24 135-6°,

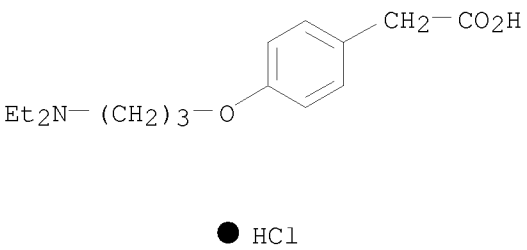
52%; m-isomer, b28 126-9°, 22%; p-isomer, b31 128-30°, n25D 1.4776, 48%. Et (4-amino-3-nitrophenyl)acetate, bright yellow, m. 80-1° (68% on saturating the acid in EtOH with HCl and standing overnight). 3,4-MeO(O2N)C6H3CH2Cl through the nitrile yields (4-methoxy-3-nitrophenyl)acetic acid, m. 122-5°. MeSPh (24.8 g.), 150 mL. CS2, and 24 g. AcCl at 0°, treated with 30 g. AlCl3 (in portions) and the mixture stirred 4 h., give p-methylmercaptoacetophenone (III), m. 72-5° 49.8 g. III, 9.6 g. S, and 27 mL. morpholine, refluxed overnight, treated with 400 mL. concentrated HCl and 300 mL. H2O, and again refluxed overnight, give 25 g. (p-methylmercaptophenyl)acetic acid, m. 92-4° Me ester b3 179-81°. m-F3CC6H4CN (51.5 g.) in 50 mL. ether, added (1 h.) to MeMgI (60 g. MeI) and, after 3 h., poured into 500 g. ice and 100 mL. concentrated HCl, gives 50% m-(trifluoromethyl)acetophenone (IV), b. 198-200°. m-F3CC6H4COCl (b750 184-6°, 95.5% yield) (93.5 g.) in 100 mL. ether, added dropwise to CdMe2 (25 mg. Mg, 100 g. MeBr, and 110 g. CdCl2) in 700 mL. ether, gives 91% IV. IV (10 g.), 2 g. S, and 5.3 g. morpholine, heated 16 h. at 135°, treated with 30 mL. AcOH and 50 mL. concentrated HCl, and refluxed 7 h., give 89% [m-(trifluoromethyl)phenyl]acetic acid, m. 72-3°. p-PhOC6H4Ac (60 g.), 13 g. S, and 10 mL. morpholine, refluxed overnight, the crude product hydrolyzed (2 days) by refluxing with 75 g. KOH in 75 mL. H2O and 600 mL. EtOH, and the acid esterified with EtOH and H2SO4, give 25 g. Et (p-phenoxyphenyl)acetate, b0.2 173-4°. p-MeOC6H4CONHC6H4CH2CO2H (m. 211-12°) and excess CH2N2 in MeOH-ether give a quant. yield of the Me ester, m. 162°. Ph2S and AcCl give p-phenylmercaptoacetophenone, b1 180°, which, by the Willgerodt method and esterification, yields Et (p-phenylmercaptophenyl)acetate, b0.65 163°. I (36 g.) in 300 mL. absolute EtOH containing 11 g. MeONa, refluxed overnight with 30 g. Et2N(CH2)3Cl, gives 24 g. Et [p-(3-diethylaminopropoxy)phenyl]acetate, b0.3 145-7° (HCl salt, m. 121°). p-HOC6H4CH2CONHCH2CH2OH (V) (49 g.) in 165 mL. 10% NaOH, treated with PhN2Cl (23 g. PhNH2) at 0°, gives 56.5 g. N-2-hydroxyethyl-α-(4-hydroxy-3-phenylazophenyl)acetamide, m. 180-1.5°. V (49 g.) and 79.7 g. Hg(OAc)2 in 800 mL. 50% EtOH and 40 mL. AcOH, allowed to stand 12 days at room temperature and the solid product heated with 750 mL. 50% EtOH containing 5% AcOH, gives 51.4 g. N-2-hydroxyethyl-α-[3,5-bis-(acetylmercuri)-4-hydroxyphenyl]acetamide, partially m. at 240° (rapid heating). p-tert-BuC6H4Ac (87 g.) through the acid (Willgerodt method), yields 19.4 g. Et (p-tert-butylphenyl)acetate, b0.47 95°. p-tert-AmC6H4Ac (68.5 g.) yields 15 g. Et (p-tert-amylphenyl)acetate, b2 124°. Reaction of I (90 g.) and 70 g. CH2:CHCH2Br, followed by esterification, gives 18.4 g. Et (p-allyloxyphenyl)acetate (VI), b0.5 126-7° oxidation of 44 g. VI in 100 mL. 70% Me2CO with 22 g. KMnO4 in 300 mL. 70% Me2CO (with addition of 8 g. AcOH to the mixture) yields 24.8 g. Et [p-(2,3-dihydroxypropoxy)phenyl]acetate, b0.2 200°. N-2-Hydroxyethyl amides, RC6H4CH2CONHCH2CH2OH, were prepared by heating the above and other esters with excess H2NCH2CH2OH overnight on the steam bath or several hrs. at 110-20° (R given; S is 1 unless otherwise given): p-acetamido m. 145-6°, p-allyloxy m. 84-5° (S 1.23), 4-amino-3-nitro m. 132°, p-NH2 m. 103-4° (S 1.14), p-tert-Am oil, p-anisoylamino m. 210-11°, 4-bromo-3-chloro m. 104-6° (S 1.71), o-Br m. 106-7°, m-Br m. 129-30° (S 2.21), p-Br m. 108-9° (S 2.90), p-tert-Bu, oil, o-Cl m. 99-100°, m-Cl m. 114-17° (S 1.84), p-Cl m. 90-1° (S 1.97), 3,5-bis(acetylmercuri)-4-hydroxy, 3,5-dibromo-4-hydroxy m.

200-2°, 3,4-di-Br m. 125-7°, 2,4-di-Cl m. 118-19°, 3,4-di-Cl m. 113-14° (S 2.10), p-(3-diethylaminopropoxy) oil, p-(2,3-dihydroxypropoxy) oil (S 1.20), 3,5-diiodo-4-hydroxy m. 179-80°, 2,3-di-MeO m. 93°, 3,4-di-MeO m. 96-8°, 3,4-di-Me m. 99-100° (S 1.27), p-EtO m. 90-1° (S 1.26), o-F m. 103-5° (S 1.23), m-F m. 75-7° (S 1.93), p-F m. 75° (S 1.54), o-HO oil (S 1.24), m-HO m. 92-3° (S 1.13), p-HO m. 110-12°, p-(2-hydroxyethylcarbamyl) m. 157-8°, 4-hydroxy-3-phenylazo m. 180-1.5°, m-I m. 127-9° (S 1.75), p-I m. 112-13° (S 1.83), 5-isopropyl-2-Me oil, p-iso-Pr oil (S 1.33), o-MeO oil, m-MeO m. 59°, p-MeO m. 86-8° (S 1.22), 3,4-methylenedioxy m. 99-100°, p-methylmercapto m. 115-17° (S 1.49), 4-methoxy-3-nitro m. 69°, o-Me m. 63-4° (S 1.36), m-Me oil (S 1.39), p-Me m. 76-8° (S 1.69), p-NO2 m. 140-2°, p-PhO m. 95° (S 1.64), p-phenylmercapto m. 89-90°, p-Ph m. 172-5° (S 0.87), 3,4,5-tri-Br m. 212-13° (S 0.33), m-F3C oil (S 1.28), 2,4,6-tri-Me m. 144-5°. N-Allyl-α-(p-hydroxyphenyl)acetamide m. 84-6°. N-(2-Aminoethyl)-α-(p-methoxyphenyl)acetamide-HCl m. 135-8° (S 1.34). PhCH2CS2Me (18.2 g.) in 15 g. MePrNH on heating to boiling gives 86% N-methyl-N-propyl-α-phenylthioacetamide, b1.5 155-8°, n24.5D 1.5876. The following phenylthioacetyl derivs. were prepared by exactly neutralizing the amino acid with 4 N NaOH, diluting with an equal volume of EtOH, adding 10% molar excess PhCH2CS2Me, and shaking for a few min. to several hrs.: D-penicillamine m. 132-3°, 55%; L-isomer m. 133-4°, 61%; β,β-diethoxyalanine, with 0.5 mol. H2O, m. 67.5-8°, 84%; DL-valine m. 102-3°, 95%; DL-isoleucine m. 95-6°, 75%. Details are given of the formation of p-HOC6H4CH2CONHCH2CH2OH. From the results of the S data it is difficult to draw any generalizations. Both the kind and position of the substituents had a marked influence upon the ability of the compound to act as a penicillin precursor. That the nature of the PhCH2CO2H derivative had a profound influence upon its utilization by the mold was illustrated in several cases.

IT 861065-20-5P, Acetic acid, [p-(3-diethylaminopropoxy) phenyl]-, hydrochloride
RL: PREP (Preparation)
(preparation of)

RN 861065-20-5 CAPLUS

CN Benzeneacetic acid, 4-[3-(diethylamino)propoxy]-, hydrochloride (1:1) (CA INDEX NAME)



L10 ANSWER 22 OF 25 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 1947:814 CAPLUS
DOCUMENT NUMBER: 41:814
ORIGINAL REFERENCE NO.: 41:155c-i,156a-i,157a-g
TITLE: Amino alcohol esters of hydroxybenzoic acids
INVENTOR(S): Christiansen, Walter G.; Harris, Sidney E.
PATENT ASSIGNEE(S): E. R. Squibb & Sons
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2404691		19460723	US	<--

GI For diagram(s), see printed CA Issue.

AB Amino alc. esters of hydroxybenzoic acids, effective for inducing local anesthesia and having the general formula in which R is a bivalent aliphatic, cycloaliph., or aromatic radical providing a continuous C bridge, R' and R'' represent alkyl, aralkyl, hydroxyalkyl, or hydroxyaralkyl, or jointly represent an alkylene group, R''' represents an aliphatic, aromatic, or araliph. radical, R'''' represents H, alkyl, or an alkoxy radical, and Y is H or alkyl, are prepared by treating an aracyl halide with an amino alc. p-EtOC6H4COCl (10 g.) in 50 cc. dry benzene is treated with 6.8 g. Et2NCH2CH2OH. A precipitate forms, and the reaction is completed by heating on the H2O bath. The solution is cooled, the precipitate is filtered and treated with a slight excess of 2 N KOH, and the ester is extracted with Et2O and dried with anhydrous Na2SO4. The Et2O solution is treated with dry HCl, and the precipitate is filtered and washed with dry Et2O to yield 2-diethylaminoethyl p-ethoxybenzoate-HCl, m. 172.5-3.5°. p-EtOC6H4COCl (4.1 g.) in 15 cc. dry benzene is refluxed 30 min. with 3.5 g. AmNEtCH2CH2OH in 10 cc. dry benzene. The benzene is distilled in vacuo and the residue is dissolved in EtOH, decolorized with C, repptd. with dry Et2O, and recrystd. from Me2CO-petr. ether to give 2-(ethylamylamino)ethyl p-ethoxybenzoate-HCl, m. 108-10°. By processes essentially similar to the above described ones were prepared 2-dibutylaminoethyl p-ethoxybenzoate-HCl, m. 144.5-5.5°; 3-dibutylaminopropyl p-ethoxybenzoate-HCl, m. 85.6-6.6°; 2-diethylaminoethyl p-butoxybenzoate-HCl, m. 146°; 2-diethylaminoethyl 2-ethoxy-3-methylbenzoate-HCl, m. 97-7.5°; 2-dimethylaminoethyl p-butoxybenzoate-HCl, m. 132-3°; 2-diethylaminoethyl o-ethoxybenzoate-HCl, m. 139-9.5°; 2-diethylaminoethyl p-(2-diethylaminoethoxy)benzoate-HCl, hygroscopic crystals, m. 143°; 2-diethylaminoethyl 2-methyl-4-ethoxybenzoate-HCl, m. 101-3°; 2-diethylamino-Et 3-methyl-4-ethoxybenzoate-HCl, m. 142.5-5°; 2-diethylaminoethyl p-(2-bromallyloxy)benzoate-HCl, m. 81.5-3.5°; and 2-diethylaminoethyl 3-methoxy-4-ethoxybenzoate-HCl, m. 171.5-2.5°. A mixture of 5.5 g. Et2NCH2CH2CH2OH, 9.3 g. p-EtOC6H4COCl and 25 cc. 10% NaOH solution is vigorously stirred 0.5 h., cooled, and extracted with benzene. The benzene solution is washed with dilute NaOH and H2O, and distilled The residual oil is dissolved in absolute alc. HCl and diluted with Et2O. The precipitate is filtered and recrystd. from EtOH-Et2O to give 3-diethylaminopropyl

p-ethoxybenzoate-HCl, m. 148.5-9.5°. 2-Diethylaminocyclohexanol (6.8 g.) in 75 cc. dry benzene is treated with 10 g. finely powdered anhydrous K2CO3 and then with 7.3 g. p-EtOC6H4COCl. The mixture is refluxed several hrs. and treated with 100 cc. H2O and 100 cc. benzene. The benzene layer is removed and purified and treated as in the above preparation to yield 2-diethylaminocyclohexyl p-ethoxybenzoate-HCl, m. 184-5°. In substantially the same manner were prepared 2-hydroxy-3-diethylaminopropyl p-ethoxybenzoate-HCl, m. 120-6°; and (N-phenacyl-N-ethylamino)ethyl p-ethoxybenzoate-HCl, white crystals. (HOCH2CH2)2NEt (6.7 g.) in 100 cc. dry benzene is treated with 14 g. anhydrous K2CO3 and then with 9.2 g. p-EtOC6H4COCl, and the mixture is refluxed with stirring for 2 h. The mixture is filtered, the benzene evaporated, and the residue distilled in vacuo to yield 2-[ethyl(2-hydroxyethyl)amino]ethyl p-ethoxybenzoate, thick, colorless oil, b8 218-25°; HCl salt, hygroscopic crystals. In similar manner were prepared 2-diethylaminoisohexyl p-ethoxybenzoate, b2.5 175-85°, b5 193-5°; 3-diethylamino-2-hydroxypropyl p-butoxybenzoate-HCl, mixture of 2 isomers, m. 79-96°; 2-[ethyl(2-hydroxyethyl)amino]ethyl p-butoxybenzoate, b3 216-20°; HCl salt, hygroscopic. A mixture of 1.5 g. Me2NCH2CEt(OH)CH2NMe2 in 5 cc. CHCl3 and 1.6 g. p-EtOC6H4CO2H in 5 cc. CHCl3 is heated 5 min. on the steam bath. Dry _ Et2O is added, and the precipitate is filtered, washed, and dried to give 1,1-bis(dimethylaminomethyl) Pr p-ethoxybenzoate-HCl, white crystalline powder, m. 121-1.5°. In like manner was prepared 1,1-bis(dimethylaminomethyl)propyl p-butoxybenzoate-HCl, m. 111°. m-EtOC6H4COCl (11.5 g.) in 50 cc. dry benzene is mixed with 14.5 Et2NCH2CH2OH in 50 cc. dry benzene, and the mixture heated on the steam bath 1 h. The precipitate is filtered, and the benzene filtrate is distilled The residue is distilled in vacuo to give 2-diethylaminoethyl m-ethoxybenzoate, b2 163-75°. This was dissolved in alc. HCl, and repptd. with Et2O to yield the HCl salt, m. 125-5.5°. Similarly were prepared 2-diethylaminoethyl p-(2-ethoxyethoxy)benzoate-HCl, m. 102-3.5°; 2-diethylaminoethyl p-propoxybenzoate, b4 160-5° (HCl salt, m. 135-6°); 2-diethylaminoethyl p-isopropoxybenzoate-HCl, m. 125.5°; and 2-diethylaminoethyl p-allyloxybenzoate, b4 165-75° (HCl salt, m. 130°). A mixture of 2.5 g. p-EtOC6H4CO2CH2CH2CH:CHBr, 5.5 g. Et2NH, and 15 cc. benzene is heated in a sealed tube at 125-35° for 8 h. After cooling, the mixture is treated with H2O and extracted with Et2O. The Et2O extract is washed with H2O, dried, and distilled on the steam bath, finally under reduced pressure. The residue is dissolved in alc. HCl and precipitated with Et2O. Washing with dry Et2O of the oily precipitate yields 4-diethylamino-4-butenyl p-ethoxybenzoate-HCl, yellowish white crystals, m. 146-7°. Heating Et2NCH2CMe2CH2OH with p-EtOC6H4COCl in dry Me2CO yields 2,2-dimethyl-3-diethylaminopropyl p-ethoxybenzoate-HCl, m. 122-4°. 3,4-Me (BuO)C6H3COCl (1.05 g.) and 1.25 g. (Me2NCH2)2C(OH)CH2CH2Ph in 10 cc. CHCl3 are refluxed for a few min., treated with dry Et2O to incipient precipitation, and allowed to stand. The crystalline precipitate which seps. after some time is filtered and washed with dry Et2O to give 1,1-bis(dimethylaminomethyl)-3-phenylpropyl 3-methyl-4-butoxybenzoate-HCl, m. 161-2°. Similarly were prepared 2,2'-bis(dimethylamino)isopropyl p-propoxybenzoate mono- and di-HCl salts, m. 208°; 3-dimethylaminopropyl 3-methyl-4-butoxybenzoate-HCl, white crystalline powder, m. 125.5-6.5°; 3-dimethylaminopropyl p-(2-phenylethoxy)benzoate-HCl, m. 156.5-7-5°; and

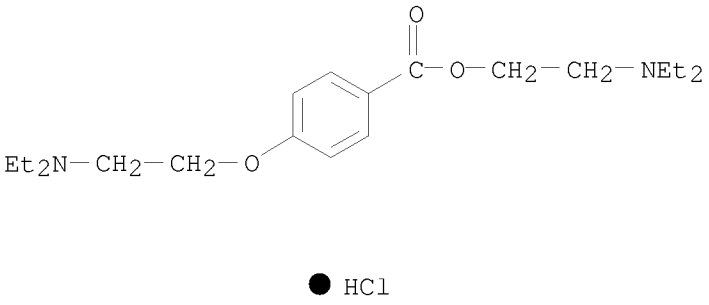
1-methyl-1-(dimethylaminomethyl)amyl 3-methyl-4-butoxybenzoate-HCl, m. 126-31°. p-EtOC6H4CO2CH2CH2NEtCH2COPh (0.9 g.) in 60 cc. EtOH containing 0.3 g. PtO is shaken 8 h. under a pressure of 35 lb. H, filtered, and the filtrate is concentrated to a small volume and diluted with Et2O. The crystalline precipitate is filtered, washed with Et2O, and dried in vacuo over CaCl2 to give 2-[ethyl(2-phenyl-2-hydroxyethyl)amino]ethyl p-ethoxybenzoate-HCl. 2-Diethylaminoethyl p-(p-aminobenzyloxy) benzoate-HCl, m. 185-7°, is prepared in the same manner, p-HOC6H4CO2CH2CH2NEt2 (0.4 g.) in 50 cc. dry Me2CO containing 15 g. anhydrous K2CO3 is treated with 5.5 g. p-O2NC6H4CH2Br, and the mixture is refluxed 12 h. The mixture is filtered, and the Me2CO distilled from the filtrate. The residue is treated with alc. HCl and diluted with Me2CO and Et2O. The precipitate is recrystd. from Me2CO-Et2O to give 2-diethylaminoethyl p-(p-nitrobenzyloxy)benzoate-HCl, m. 145-6°. In addition, 21 other similar compds. are cited, but no phys. properties are recorded. The prepns. of many intermediates used in preparing the above compds. are described. A solution of 3.5 g. Na in 100 cc. absolute EtOH is treated first with 25 g. 2,3-HO(MeO)C6H3CO2Et and then with 20 g. EtBr, and the solution is boiled until neutral to moist litmus. The mixture is filtered, and the EtOH is removed from the filtrate. The residue is fractionated to yield Et 2-ethoxy-3-methylbenzoate, b6 116-18°, which upon hydrolysis with alc. NaOH yielded 2-ethoxy-3-methylbenzoic acid, oily precipitate, which was extracted with ether. The ether was removed and the residue treated with SOCl2 to give 2-ethoxy-3-methylbenzoyl chloride, b2.5 102-5°. p-(2-Phenylethoxy)benzoic acid, white powder, m. 163-4° (chloride, b5 215-30°), and 3-methyl-4-(2-phenylethoxy) benzoic acid, m. 150-2° (chloride, b1 210-15°), were prepared in essentially the same manner. p-HOC6H4CO2Me (13 g.) in 35 cc. Me2CO is treated with 15 g. anhydrous K2CO3, the mixture is refluxed and stirred, treated with 13 g. Et2NCH2CH2Cl, heated, stirred 15 h., filtered, and the filtrate concentrated by distillation The residue is treated with excess NaOH and boiled until saponification is complete. The solution is extracted with Et2O, and the aqueous solution is evaporated to dryness in vacuo. The residue is extracted with absolute EtOH, the extract filtered, the filtrate evaporated to dryness, and the residue recrystd. from MeOHEt2O to give p-(2-diethylaminoethoxy)benzoic acid-HCl, white needles, m. 160-1°. Treatment with PCl5 yields p-(2-diethylaminoethoxy)benzoyl chloride-HCl, m. 143°. In similar manner were prepared 2-methyl-4-ethoxybenzoyl chloride, colorless liquid, b3 138-40°; 3-methyl-4-ethoxybenzoyl chloride, colorless liquid, b6 147-52°; p-(2-ethoxyethoxy)benzoic acid, m. 131-2° (chloride, b5 150-60°); p-(2-bromoallyloxy)benzoic acid, m. 200° (decomposition) (chloride, b5 160-70°); 3-methoxy-4-ethoxybenzoyl chloride, b5 147-50°, m. 72°, and 3-methyl-4-butoxybenzoic acid, white plates from 60% EtOH, m. 144-6° (chloride, b1.5 144-54°). A mixture of 5.5 g. dry p-EtOC6H4CO2Na, 8 g. BrCH:CHCHBrMe, and 10 g. dry xylene is heated in a sealed tube at 165-70° for 6 h. The contents of the tube are extracted with dilute EtOH and Et2O. The Et2O is washed with H2O, dried over Na2SO4, and distilled The oily residue is fractionated in a high vacuum to yield 3-bromo-1-butenyl p-ethoxybenzoate, b3 165-75°. A mixture of 9.95 g. PhCOCH2Cl, 4.4 g. EtNHCH2CH2OH, and 100 cc. benzene is refluxed 3 h. On

adding 10 g. K_2CO_3, a vigorous evolution of CO_2 ensues. The suspension is further refluxed 4 h. and filtered. The filtrate is treated with HCl in Et_2O. The reddish brown semisolid which seps. is filtered, washed with Et_2O, and dried in a vacuum over $CaCl_2$ to yield the very hygroscopic N-phenacyl-N-ethyl-2-aminoethanol-HCl, which is treated with p-$EtOC_6H_4COCl$ in benzene in the presence of K_2CO_3 in the regular manner to give N-phenacyl-N-ethyl-2-aminoethyl p-ethoxybenzoate-HCl, white crystals.

IT 855470-53-0P, Benzoic acid, p-(2-diethylaminoethoxy)-, 2-diethylaminoethyl ester, hydrochloride
RL: PREP (Preparation)
(preparation of)

RN 855470-53-0 CAPLUS

CN Benzoic acid, 4-[2-(diethylamino)ethoxy]-, 2-(diethylamino)ethyl ester, hydrochloride (1:1) (CA INDEX NAME)



L10 ANSWER 23 OF 25 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 1938:59916 CAPLUS
DOCUMENT NUMBER: 32:59916
ORIGINAL REFERENCE NO.: 32:8391e-h
TITLE: The relation between chemical constitution and local-anesthetic activity. II. Some alkoxybenzoates of di-alkylamino alcohols
AUTHOR(S): Lott, W. A.; Harris, S. E.; Christiansen, W. G.
SOURCE: Journal of the American Pharmaceutical Association (1912-1977) (1938), 27, 661-5
CODEN: JPHAA3; ISSN: 0003-0465
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

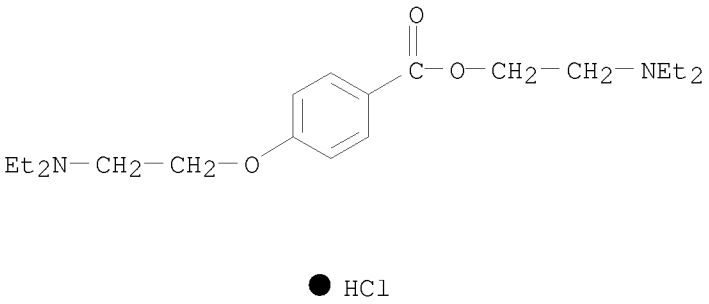
AB The HCl salts of the diethylaminoethyl esters of the following alkoxybenzoic acids were prepared: p-methoxy, m. 142°; p-ethoxy, m. 177.3°; p-propoxy, m. 137.6-8.1°; p-isopropoxy, m. 125.5°; p-butoxy, m. 146.5-7.5°; p-allyloxy, m. 130°; p-β- phenylethoxy, m. 91-2°; p-β-ethoxyethoxy, m. 102-3.5°; p-β-bromoallyloxy, m. 81.5-3.5°; p-β-diethylaminoethoxy, hygroscopic; o-ethoxy, m. 139-9.5°; m-ethoxy, m. 125-5.5°. The p-ethoxybenzoic ester HCl salts of the following alkylamino alcs. were prepared: ethylamylaminoethyl, m. 108-10°; β-dibutylaminoethyl, m. 144.5-5.5°; γ-dibutylaminopropyl, m. 85.5-6.5°; β,β-dimethyl-γ-diethylaminopropyl, m. 121-1.5°; γ-diethylaminopropyl, m. 149.9-50.4°; β-diethylamino-δ-

methylamyl, oil; α,α-bis(dimethylaminomethyl)propyl, m. 121-1.5°; α-methyl-α-diethylaminomethylpropyl, m. 122-4°; β-diethylaminoethoxyethyl, m. 112-15°; 2-diethylaminocyclohexyl, m. 184-5°; 1-diethylamino-2,3-propanediol, m. p. indefinite; N-ethyldiethanolamine, oil. The p-butoxybenzoic ester HCl salts of the following alkylamino alcs. were prepared; N-ethyldiethanolamine, m. 79.6°; 1-diethylamino-2,3-propanediol, m. p. indefinite; β-dimethylaminoethyl, m. 132-3°. These compds. all proved to be local anesthetics in preliminary pharmacol. tests, details of which will be published shortly.

IT 855470-53-0P, Benzoic acid, p-(2-diethylaminoethoxy)-, 2-diethylaminoethyl ester, -HCl
RL: PREP (Preparation)
(preparation of)

RN 855470-53-0 CAPLUS

CN Benzoic acid, 4-[2-(diethylamino)ethoxy]-, 2-(diethylamino)ethyl ester, hydrochloride (1:1) (CA INDEX NAME)



L10 ANSWER 24 OF 25 CAPLUS COPYRIGHT 2008 ACS on STN
ACCESSION NUMBER: 1934:60903 CAPLUS
DOCUMENT NUMBER: 28:60903
ORIGINAL REFERENCE NO.: 28:7429h-i,7430a-b
TITLE: Dialkylaminoalkyl esters of hydroxy-3-carboxybiphenyls
INVENTOR(S): Christiansen, Walter G.; Harvey, Adelbert W.
PATENT ASSIGNEE(S): E. R. Squibb & Sons
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 1976922		19341016	US	<--

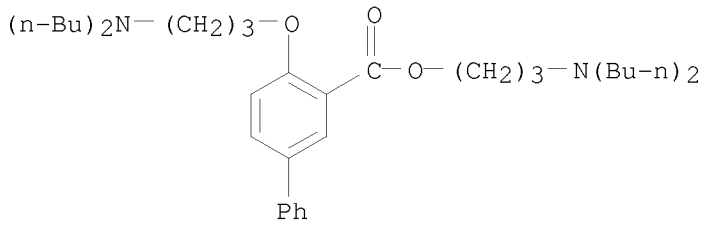
AB Compds. (suitable for use as local anesthetics in solution buffered with a phosphate) such as the dialkylaminoalkyl esters of 3 - carboxy - 4 - hydroxybiphenyl and 3 - carboxy - 2-hydroxybiphenyl and salts thereof, particularly 3-β-diethylaminocarbethoxy-4-hydroxybiphenyl and its salts are prepared by converting the hydroxy-3-carboxybiphenyl to a salt, forming a halide ester, preferably a bromoalkyl ester from the salt and then forming the dialkylaminoalkyl ester from this. Purification of the 3-β-diethylaminocarbethoxy-4-hydroxybiphenyl hydrochloride may be

accomplished by crystallization from absolute EtOH. The product, in the form of the hydrochloride, is a white crystalline substance soluble in water, m. 167-168.5°. The free ester is an almost colorless oil. Starting with 3-carboxy-2-hydroxybiphenyl and employing similar reactions, corresponding alkyl derivs. may be formed in which the hydroxy group is in the 2- instead of the 4-position.

IT 873986-35-7, Benzoic acid, 2-(γ-dibutylaminopropoxy)-5-phenyl-, γ-dibutylaminopropyl ester (and salts)

RN 873986-35-7 CAPLUS

CN [1,1'-Biphenyl]-3-carboxylic acid, 4-[3-(dibutylamino)propoxy]-, 3-(dibutylamino)propyl ester (CA INDEX NAME)



L10 ANSWER 25 OF 25 CAPLUS COPYRIGHT 2008 ACS on STN

ACCESSION NUMBER: 1934:60902 CAPLUS

DOCUMENT NUMBER: 28:60902

ORIGINAL REFERENCE NO.: 28:7429g-h

TITLE: Dialkylaminoalkyl esters of dialkylaminoalkoxy-3-carboxybiphenyl

INVENTOR(S): Christiansen, Walter G.; Braker, William

PATENT ASSIGNEE(S): E. R. Squibb & Sons

DOCUMENT TYPE: Patent

LANGUAGE: Unavailable

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

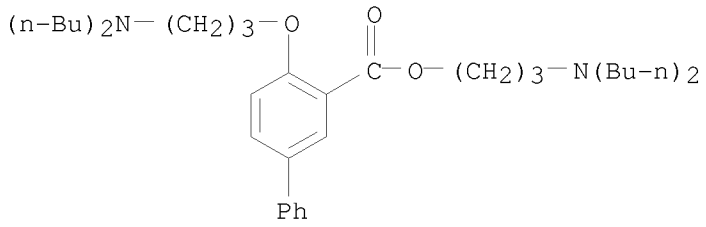
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 1976921		19341016	US	<--

AB Compds. (suitable for use in the preparation of local anesthetics) such as 3-β-diethylaminocarbethoxy-4-β-diethylaminoethoxybiphenyl and 3-γ-dibutylaminocarbopropoxy - 4 - γ - dibutylaminopropoxybiphenyl are prepared from a hydroxy-3-carboxybiphenyl by forming its di-Na derivative and then replacing the Na atoms by dialkylaminoalkyl radicals (various details for preparing these compds. and their hydrochlorides and borates being given).

IT 873986-35-7, Benzoic acid, 2-(γ-dibutylaminopropoxy)-5-phenyl-, γ-dibutylaminopropyl ester (and salts)

RN 873986-35-7 CAPLUS

CN [1,1'-Biphenyl]-3-carboxylic acid, 4-[3-(dibutylamino)propoxy]-, 3-(dibutylamino)propyl ester (CA INDEX NAME)


(n-Bu)2N— (CH2)3— O
O
C— O— (CH2)3— N(Bu-n)2
Ph